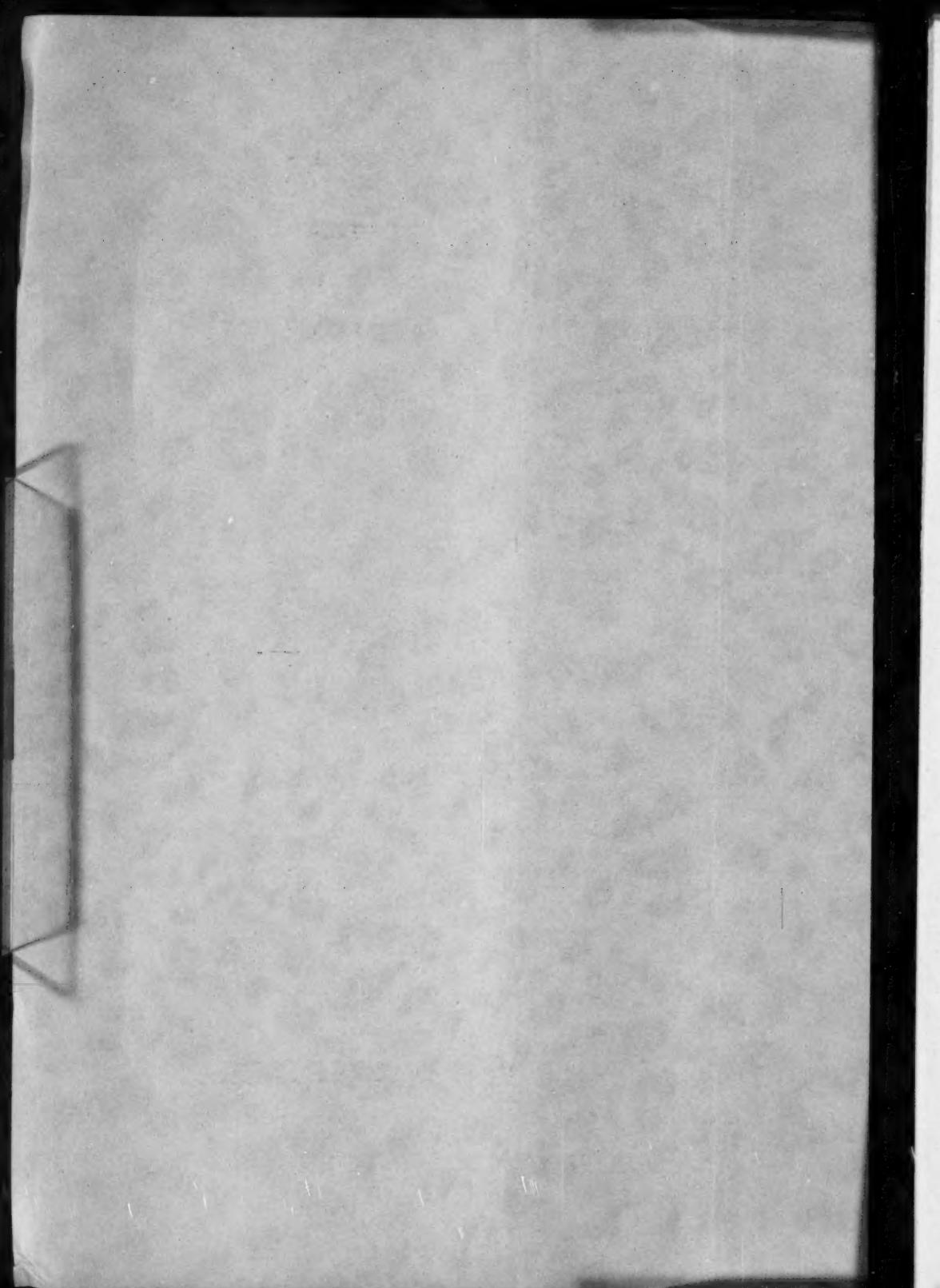


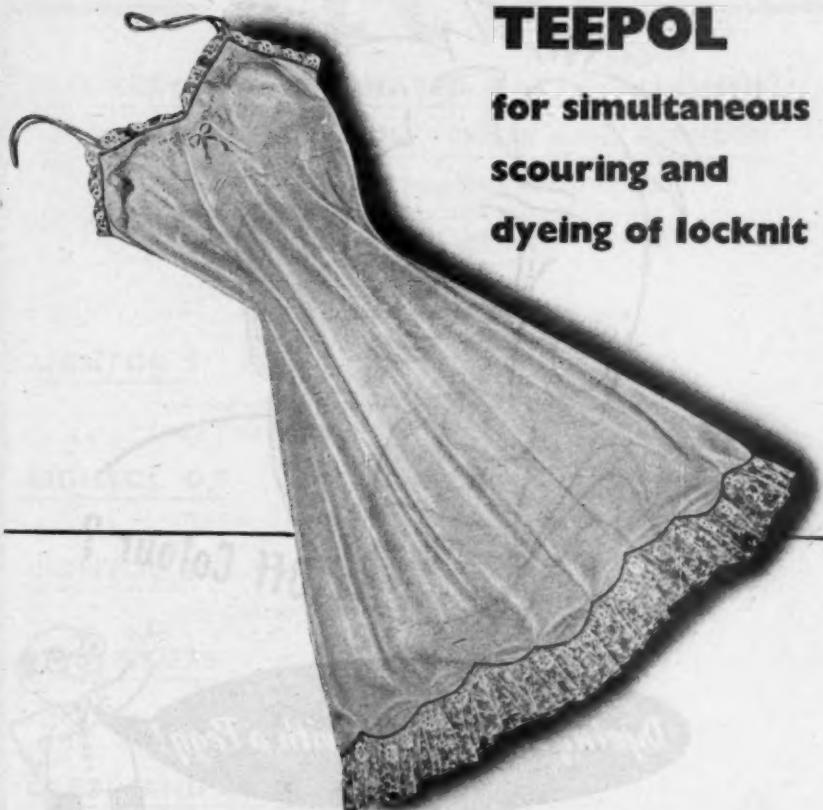
# The Journal *of the* SOCIETY OF DYERS AND COLOURISTS

Volume 67 Number 1

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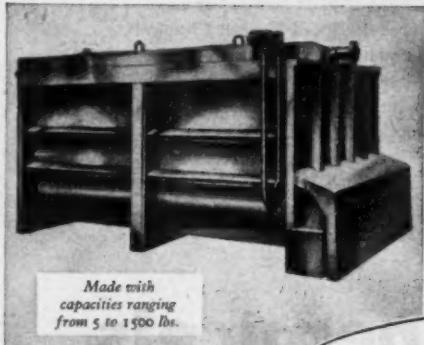
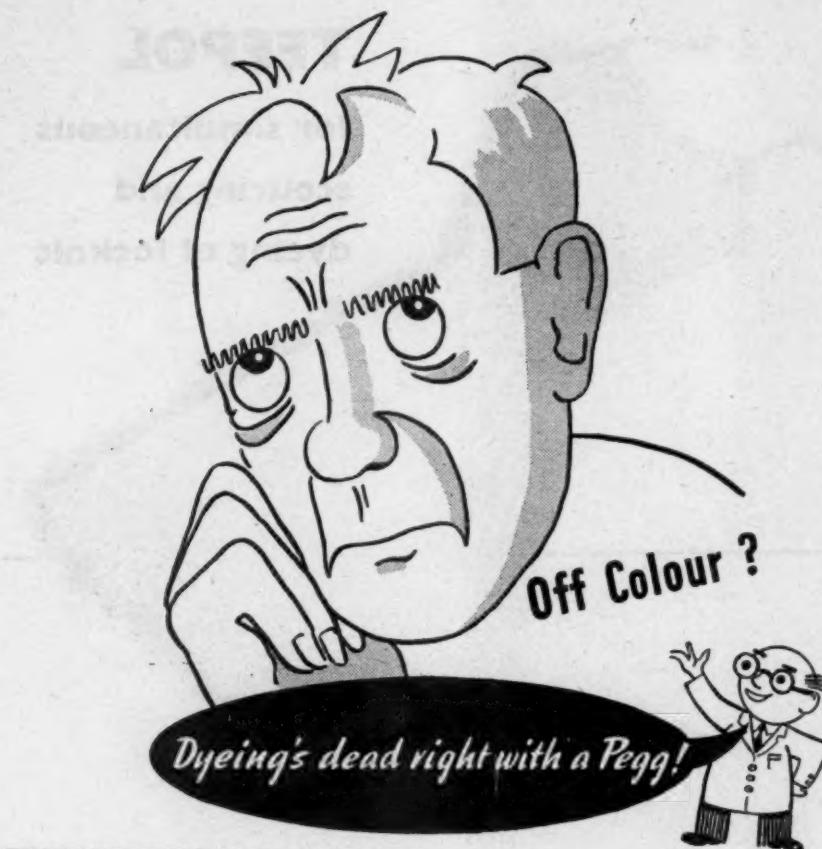
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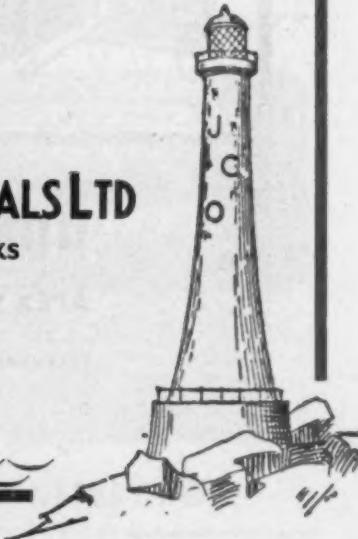
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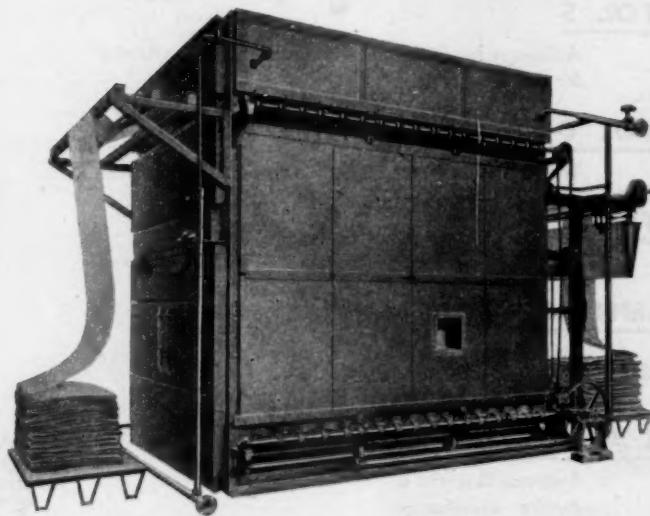
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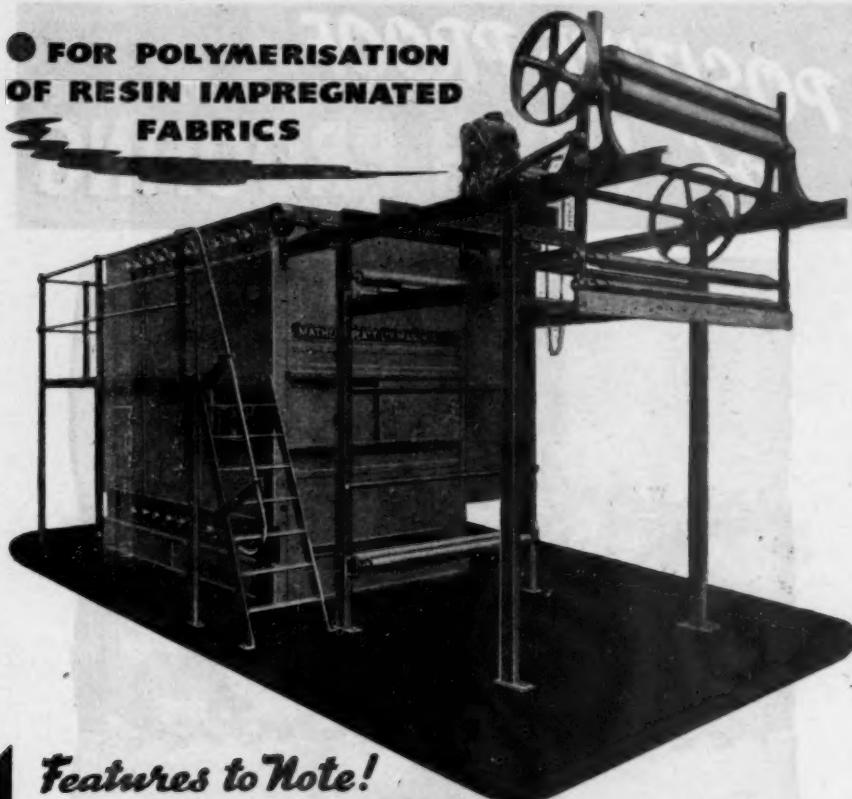
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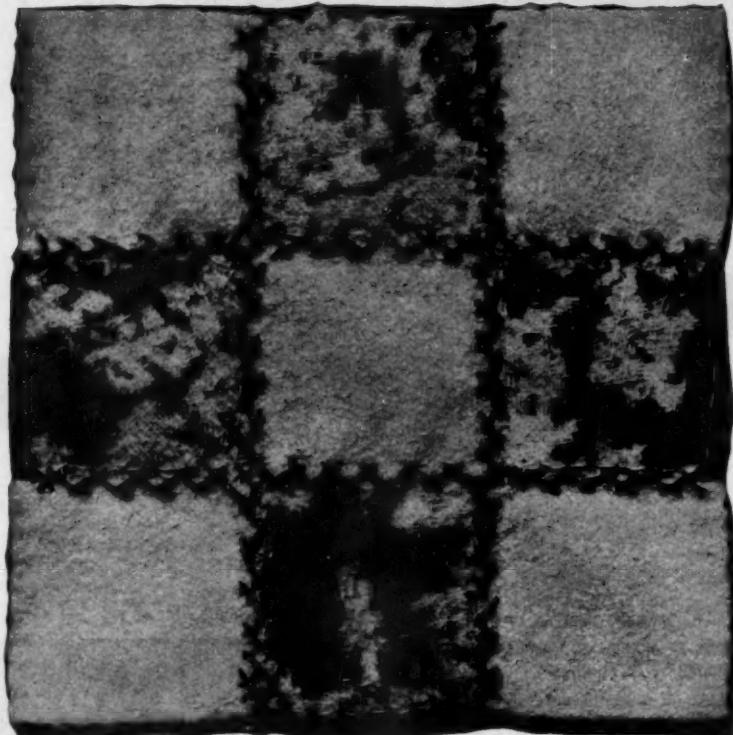
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## NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1-6 of the January 1951 and pages 345-348 of the July 1950 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, 32-34 Piccadilly, Bradford, Yorkshire (Telephone Bradford 25138-9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

## Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

### LECTURE

Some Applications of the Report of the Committee on the Dyeing Properties of Wool Dyes *B. Kramisch*

### COMMUNICATIONS

Some Observations on the Hardening Process used in Felt Manufacture. I—Factors influencing Hardening *R. S. M. Frohnsdorff and C. S. Whewell*

The Reaction of Acrylonitrile with Macromolecular Hydroxy Substances—

I—General Review *J. H. MacGregor*

II—The Production and Uses of Water-insoluble, Alkaline-soluble Cyanoethyl Cellulose Ethers as Soluble Yarns *J. H. MacGregor and C. Pugh*

Combination of Wool with Acids in presence of Salts *B. Olofsson*

Some Aspects of the Drying and Heating of Textiles—

IV—The Effect of Drying Conditions on the Latent Strain in the Yarn of Viscose Cakes *J. M. Preston and J. C. Chen*

V—Migration in relation to Moisture Content *J. M. Preston and A. Bennett*

Studies in the Fundamental Processes of Textile Printing. II—The Influence of Thickener Composition on Dye Transfer during Steaming *H. A. Turner and D. A. Reilly*

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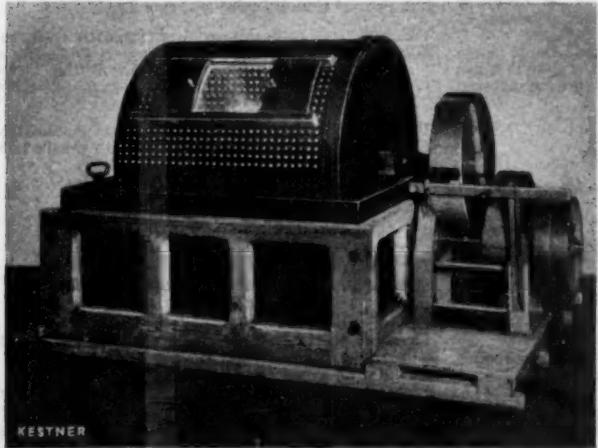
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## Forthcoming Meetings of the Society

1951	
Friday 30th Mar.	ANNUAL GENERAL MEETING AND DINNER OF THE SOCIETY, GRAND HOTEL LEICESTER
Friday 20th April	EIGHTH MERCER LECTURE—Dr. D. Traill, St. Enoch Hotel, Glasgow

### HUDDERSFIELD SECTION

All meetings held at Field's Cafe, Westgate, Huddersfield, at 7.30 p.m. unless otherwise stated

1951					
Tuesday 27th Feb.	H. W. Ellis, Ph.D., A.R.C.S., A.R.I.C. <i>The Ellis Photometer</i> F. L. Warburton, Esq. (W.I.R.A.) <i>Artificial Daylight Lamps</i>	Wednesday 14th Feb.	W. J. Watts, Esq. (Aridye Ltd.) <i>Resin-bonded Pigments</i>		
Tuesday 20th Mar.	F. Jackson, Esq. <i>Water Treatment for Boilers and Processing</i>	Wednesday 1st March	E. W. Goodale, Esq. (Warner & Son Ltd.) <i>Design in Furnishing Fabrics</i> (This will be a joint meeting with the Textile Institute)		
Tuesday 10th April	ANNUAL GENERAL MEETING Miss Esmee Smith. <i>Union Dyeing with Special Reference to Modern Fibres</i>	March	Annual General Meeting and Dinner (Date to be announced later)		

### LONDON SECTION

All meetings held in the Rooms of the Royal Society, Burlington House, Piccadilly, London W.1, at 6 p.m. unless otherwise stated

1951					
Friday 2nd Feb.	J. Bromley, Esq., and A. Cheek, Esq. (Courtaulds Ltd.). <i>The Dyeing and Finishing of Rayon Warp Knitted Fabric</i>	Thursday 8th Feb.	7.30 p.m. By invitation of the Textile Institute at The Scottish Woollen Technical College, Galashiels. Dr. P. W. Cunliffe. <i>Package Dyeing</i>		
Friday 2nd Mar.	H. A. Turner, Esq. (Manchester College of Technology). <i>A Survey of the Results of Recent Research in Dyeing and Finishing</i> .	Tuesday 20th Feb.	I. Gailey, B.Sc. <i>An investigation into the Unlevel Dyeing of Cotton Threads</i>		
	It is also intended to hold a Dinner and Dance in the New Year, and to arrange for a lecture to be given in Luton in the Spring, on a subject of interest to the hat industry.	Tuesday 27th Feb.	G. L. Boardman, Esq. (Standfast Dyers and Printers Ltd.). <i>Developments in Continuous Dyeing</i> . (Joint meeting with the Textile Institute), School of Economics, Bell Street, Dundee. 7.30 p.m.		
		Tuesday 20th March	ANNUAL GENERAL MEETING. Followed by ordinary meeting at 7.30 p.m. Details to be announced later.		

### MIDLANDS SECTION

All meetings commence at 7 p.m. (except where stated otherwise)

1951					
Wednesday 24th Jan.	COLLOQUIUM. Short papers on practical problems by local members King's Head Hotel, Loughborough	Thursday 25th Jan.	1951		
Wednesday 14th Feb.	E. J. Davies, B.Sc., M.Sc. <i>The Reactions of Fabrics and Finishes to Dry-cleaning</i> (Joint meeting with the Textile Institute) College of Technology, Leicester	Thursday 8th Feb.	Prof. J. B. Speakman. <i>Research on the Assessment of "Quality" in Wool</i>		
Friday 23rd Feb.	MIDLANDS SECTION DINNER King's Head Hotel, Loughborough	Thursday 22nd Feb.	John Boulton, M.Sc. <i>Rayon Staple—A Dyeing Miscellany</i>		
Wed. 21st Mar. 7.15 p.m.	<i>Recent developments in fluorescent lamps, with particular regard to colour problems</i> . Dr. S. T. Henderson (Joint meeting with British Association of Chemists). School of Arts and Crafts, Derby	Thursday 8th Mar.	Dr. R. L. Elliott (Bradford Technical College) <i>Modern Methods for the Investigation of Dye-stuffs</i>		
Wednesday 18th April	ANNUAL GENERAL MEETING OF SECTION King's Head Hotel, Loughborough	Thursday 22nd Mar.	Dr. G. H. Lister (Sandoz Products Ltd). <i>pH in Practical Dyeing</i>		
		Thursday 29th Mar.	J. A. Potter, M.A. (C.A.C.). <i>Dyeing for the Hosiery Trade</i>		
			ANNUAL GENERAL MEETING		

Continued on page xxix

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# THE JOURNAL OF THE Society of Dyers and Colourists

Volume 67—Number 1

JANUARY 1951

*Issued Monthly*

## OFFICIAL NOTICES

### ELECTION OF OFFICERS AND MEMBERS OF COUNCIL

Under the revised Bye-Laws which are now operative, Officers and Members of Council—other than the President and President-elect, who are nominated by Council and elected at the Annual General Meeting—are to be elected by a Postal Ballot of the members.

The attention of members is drawn to Bye-Laws No.

12-17 inclusive and 20-26 inclusive, which relate to Officers and Members of Council respectively.

Nominations to be valid must be received by the Honorary Secretary of the Society at least six weeks prior to the date of the Annual General Meeting, which is on 30th March 1951.

### MEMBERS AND JUNIOR MEMBERS

Persons desirous of joining the Society as Ordinary Members or Junior Members may obtain Application Forms from the General Secretary or from the Honorary Secretary of any Section of the Society. Applications

must be proposed and seconded by members to whom the applicants are known personally. The annual subscription is £2 2s. 0d. for Ordinary Members and 10s. 6d. for Junior Members (below 21 years of age), payable on January 1st.

### The JOURNAL

The *Journal* is posted free to all Members and Junior Members. Non-members can obtain copies on application to the Offices of the Society. All orders must be accompanied by a remittance of 5s. 0d. per copy (£3 0s. 0d. per annum post free). Back numbers of most issues of the Society's *Journal* can be supplied at the same rate.

Members are reminded that under Bye-Law 40 the *Journal* will not be forwarded to those who have not paid their subscription by 30th June 1950.

Members residing abroad are particularly requested to inform the General Secretary by separate post when sending their subscriptions by Money Order.

Communications on any subject related to the objects of the Society, especially such as are of an original character, are invited for consideration for publication in the *Journal*. Such communications in the first instance should be addressed to the Editor, Society of Dyers and Colourists, 32-34 Piccadilly, Bradford, Yorks.

General communications, including inquiries or orders for advertisements, should be addressed to the Offices of the Society, to which address all remittances should be sent.

#### REPRINTS OF LECTURES AND COMMUNICATIONS

Reprints of all lectures and communications are available after publication to members and non-members of

the Society. The charges (postage included) are as follows—Single copies, 1s. 0d. each; per dozen copies up to and including 8 pages, 7s. 6d., and for papers occupying more than 8 pages of the *Journal*, 10s. Orders should be addressed to "The Society of Dyers and Colourists, 32-34 Piccadilly, Bradford". They can be accepted only if accompanied by remittance and if received immediately after publication of the paper.

#### ABSTRACTS SECTION

Attention is drawn to the fact that copies of the Abstracts Section of the *Journal* printed on only one side of the paper, so that individual abstracts can be cut out and pasted on cards, are available at a charge of 20s. 0d. per annum. Orders should be sent to the Society's offices at 32-34 Piccadilly, Bradford, Yorks.

#### FREE ADVERTISEMENTS

For the convenience of members the Publications Committee allows a limited number of advertisements relating to SITUATIONS WANTED to be inserted in the *Journal* gratis. Such advertisements must not exceed 24 words in length.

Replies may be addressed, Box—, Society of Dyers and Colourists, 32-34 Piccadilly, Bradford, Yorks.

### NOTICE TO AUTHORS OF PAPERS

The MSS. of all papers communicated to or read before the Society become the property of the Society. They should be addressed to the Editor, Society of Dyers and Colourists, 32-34 Piccadilly, Bradford, and should be accompanied by the "Schedule of Particulars", a copy of which may be obtained from the Honorary Secretary of any Section. Authors must not allow their papers to be published elsewhere before they have appeared in the Society's *Journal*. Should prior publication take place without the sanction of the Publications Committee, the paper will be printed only as an abstract or summary.

Manuscripts submitted for publication in the *Journal* should be typewritten (double spacing) on good-quality paper, using one side of the paper only and leaving a

margin at least 1 in. wide on the left-hand side. The time taken in refereeing papers (both lectures and communications) will be reduced to a minimum when authors submit two copies of the typescript. Authors are requested to be as concise as possible. When experimental procedure has already been published, a literature reference to the paper containing the details is sufficient, whilst well known experimental methods should be described very briefly.

Introductory paragraphs describing the aims of the investigation and the method of attack are desirable, and should be followed by the experimental results and their discussion. There should be a brief summary for insertion at the beginning of the paper. Centre heading should be employed sparingly. Side headings should be indented

and underlined, and run into the text to which they apply by means of a dash. References to the literature should be numbered consecutively, using superscript numbers without brackets immediately following the text words or author's name to which they refer.

The list of references should be given at the end of the manuscript and the abbreviations used should be, as far as possible, those given in the "List of Periodicals Abstracted" included at the end of the Index to the preceding year's *Journal*. Reference numbers in this list should be neither enclosed in brackets, nor followed by full-stops. As far as possible throughout the manuscript the abbreviations listed in the Jan. 1950 issue (p. 54) should be used. Tables should be numbered consecutively in Roman numerals and Figures in Arabic numerals.

Figures and graphs should be carefully prepared,

preferably in Indian ink, on plain white drawing paper or, preferably, Bristol board. In graphs, the frame and actual curves should be ruled and inked more heavily than any co-ordinate lines, and the latter should not be close together as in ordinary graph paper. Experimental points should always be given, and they should be indicated by small circles rather than by crosses in the case of a single graph, but where several graphs appear in a single Figure clear means of differentiation must be adopted. All numbers and legends are set up in type by the printer, and authors should therefore indicate them lightly in pencil.

Twenty-five free copies of a reprint are supplied to the author of an original paper published in the *Journal*, or fifty free copies are supplied when there are two or more authors, and a further number may be purchased from the Society at the rates given above.

#### COPYRIGHT NOTICE

Original Articles, Papers, and Communications printed in this *Journal* are copyright. Subject to full acknowledgment being made, up to, but not exceeding, 800 words may be published elsewhere, but application for permission to reprint in *extenso* should be addressed to the Editor of the *Journal* at the Offices of the Society.

Lecturers are required to sign an agreement regarding copyright and publication, copies of which are supplied by the Honorary Secretaries of Sections.

The Society subscribes to the Royal Society Fair Copying Declaration (J.S.D.C., 66, 54 (Jan. 1950)).

#### LIBRARY OF THE CHEMICAL SOCIETY—LOAN OF BOOKS

Members of the Society may borrow books from the very extensive collection of works on pure and applied chemistry in the Library of the Chemical Society, by applying direct to *The Librarian, Chemical Society*.

Burlington House, London, W.I., and referring to their membership of the Society of Dyers and Colourists. Letters on the subject should not be addressed to the Offices of the Society in Bradford.

#### DEPOSIT OF SEALED COMMUNICATIONS

I.—The Society is prepared to receive from members and others and to keep as deposits, Sealed Communications dealing with any subject relating to the theory or practice of the Dyeing, Printing, and kindred industries.

II.—Every deposit must bear on the cover a title for classification, the author's name, the date, and must be secured by a distinctive seal.

III.—The deposit should be written in English, and, if it deals with machinery, be accompanied by sketches or drawings, or, in the case of dyeing or printing processes, by patterns.

IV.—Every deposit will, unopened, be signed and sealed by the Honorary Secretary of the Society, immediately after receipt, and countersigned by another member of the Council.

V.—The deposits will be numbered and entered into a

special register, and an official receipt will be sent to the author.

VI.—No charge will be made for registration and deposit.

VII.—The deposits will be kept by the Society for a period of seven years. At the end of this period they will be opened and the contents read before the next meeting of the Council of the Society, and the contents, or an abstract thereof, may be published in the *Journal* of the Society at the discretion of the Publications Committee.

VIII.—The author shall have the right to recall his deposit unopened at any time within the said seven years, or he may, at any time, order it to be opened and read before one of the Society's meetings. Such instructions must be accompanied by the official receipt.

IX.—The Society will take every reasonable care of the deposits, but cannot be held responsible for their loss.

## MEDALS AND AWARDS

## HONORARY MEMBERS OF THE SOCIETY

1886-1915	*Sir H. E. Roscoe, M.P.	1931-1933	*Alfred Rée, Ph.D.
1902-1926	*F. H. Bowman, D.Sc., F.I.C., F.R.S.E.	1932-1940	*W. M. Gardner
1908-1916	*Prof. Dr. C. Graebe	1934-1947	*George Douglas
1908-1915	*Prof. Dr. C. Liebermann	1934-1946	*H. Grandage
1911-1916	*Prof. Adolf von Baeyer	1934-1940	*Christopher Rawson
1914-1924	*Count Hilaire de Chardonnet	1941-1948	*Prof. E. C. C. Baly, C.B.E., M.Sc., F.R.S.
1917-1941	*Arthur G. Green, M.Sc., F.I.C., F.R.S.	1944-1946	*Prof. F. M. Rowe, D.Sc., F.R.I.C., F.R.S.
1919-1938	*R. Vidal	1946-1948	*James S. Ridsdale
1921-1936	*Horace A. Lowe	1947	C. J. T. Cronshaw, D.Sc., F.R.I.C., M.I. Chem.E., F.T.I., F.R.S.E.
1923-1935	*Charles F. Cross, F.R.S.	1948	H. Jeannion, M.C., A.M.C.T., A.R.I.C.
1925-1927	*Maurice Prud'homme	1950	George E. Holden, C.B.E., M.Sc., F.R.I.C.
1927-1930	*Ernest Hickson		
1928-1939	*R. E. Schmidt		

The following have been *ex officio* Honorary Members of the Society, the later dates indicating termination or change in title of the office—

1886-1920	The Worshipful Master of the Dyers' Company	1886-1900	The President, Bradford Technical College
1920-	The Prime Warden of the Worshipful Company of Dyers	1900-1905	*W. E. B. Priestley, Chairman of the Technical Instruction Committee of the Bradford City Council

\*Deceased

## THE PERKIN MEDAL

The Perkin Medal was modelled by the late F. W. Pomeroy, R.A., for the Society. It is an excellent presentation of the head of Sir William Perkin, the founder of the coal-tar colour industry, and President of the Society in 1907. This medal is awarded for discoveries or work of outstanding importance in connection with the tinctorial arts.

## LIST OF RECIPIENTS

1908	Professors Graebe and Liebermann. "Synthesis of Alizarin."
1911	Prof. Adolf von Baeyer. "Synthesis of Indigo."
1914	Comte Hilaire de Chardonnet. "Artificial Silk."
1917	Prof. Arthur G. Green. "Primuline."
1919	R. Vidal. "Sulphur Black."
1921	Horace Lowe. "Permanent Lustre on Cotton."
1923	Chas. F. Cross. "Discovery of Viscose."
1925	M. Prud'homme. "Aniline Black and Alizarin Blue."
1928	Dr. Robert E. Schmidt. "For Epoch-making Discoveries of Anthraquinone Derivatives and Dyestuffs therefrom."
1938	Dr. H. Dreyfus. "For Discoveries and Work of Outstanding Importance in Connection with the Development of the Cellulose Acetate Rayon Industry in England."
1938	J. Baddiley. "In recognition of his National Services for the Renaissance of the British Dyestuffs Industry through Many Important Investigations in the Field of Colour Chemistry Conducted or Directed by him."
1950	Prof. J. B. Speakman. "In recognition of his Outstanding Contributions to the Science and Technology of Textiles."



## THE MEDAL OF THE SOCIETY OF DYERS AND COLOURISTS

This Medal was instituted by the Society in 1908. Of the competitive designs submitted, that of Mr. Edgar Lockwood was selected. The design indicates that the work of the dyer (centre figure) is both a science (right background) and an art (left foreground).

1908-1927 The Medal was occasionally awarded as a recognition of work of exceptional merit carried out under the Society's Research Scheme.

From 1928 The Medal was awarded as a recognition of exceptional services (a) to the Society or (b) in the interests of the Tinctorial and Allied Industries.

1928 Ernest Hickson (Gold Medal). "Exceptional Services rendered to the Society as Chairman of the Publications Committee 1897-1925, and Chairman of Colour Index Committee."

1930 Arthur Silverwood (Gold Medal). "Exceptional Services rendered to the Society as Honorary Secretary 1913-1930."

## LIST OF RECIPIENTS

1908	J. B. Fothergill (Bronze Medal). "Treatment of Cotton to Cause it to Resist Direct Dyeing Colours."
1912	J. H. Garner (Silver Medal). "Treatment of Effluents from Dyehouses and Textile Factories."

- 1933 Prof. Walter M. Gardner (Gold Medal). "Distinguished Services as Editor of the Society's *Journal* 1900 to 1932."
- 1934 Prof. F. M. Rowe (Gold Medal). "Exceptional Services to the Society and to the Tinctorial Industries as Editor of the *Colour Index* 1924 and the 'Supplement' 1928."
- L. A. Lantz (*Chairman*), H. H. Bowen, P. W. Cunliffe, R. S. Horsfall, Prof. B. A. McSwiney, C. C. N. Vass, C. M. Whittaker, S. G. Barker (Silver Medals). "Exceptional Services to the Society as Members of the Standardization of Fastness Executive Committee."
- 1936 W. A. Edwards (Silver Medal). "Valuable Services rendered to the Society as Honorary Secretary of the Midlands Section since its inception in 1919 to 1935."
- 1937 R. Ritchie (Silver Medal). "Devoted and Valuable Services as Honorary Secretary of the Scottish Section for eighteen years."
- 1940 F. L. Goodall (Gold Medal). "Valuable Services to the Tinctorial Industries by his work on the Theory and Practice of Wool Dyeing."
- 1943 C. M. Whittaker (Gold Medal). "In recognition of Exceptional Services in promoting Scientific and Technical Knowledge amongst Textile Colourists, and for Sustained and Outstanding Service to the Society."
- 1946 H. H. Bowen (Gold Medal). "For Outstanding Services to the Society and in recognition of his Chairmanship of the Publications Committee for a period of twenty years."
- H. H. Hodgson (Gold Medal). "For Outstanding Services to the Society and for his series of Sustained Experimental and Theoretical Contributions to those chapters of Organic Chemistry which are the essential scientific background to the Dyestuff Industry."
- E. Race (Silver Medal). "For his Valued Services to the Society as Joint Author of thirteen papers published in the Society's *Journal*."
- H. Turner (Silver Medal). "In recognition of his Valuable Services to the Society and of his twenty-two years' service as Honorary Secretary of the Huddersfield Section."
- 1947 Fred Smith (Gold Medal). "For Exceptional Services to the Society over a period of thirty-seven years comprising Chairman, West Riding Section; Member of Council and of many important Committees."
- F. Scholefield (Gold Medal). "In recognition of Exceptional Services to the Society in the advancement of Tinctorial Technology both in theory and practice."
- C. Schardt (Silver Medal). "In recognition of his Valuable Services to the Society over a period of twenty-four years including Chairman, Vice-Chairman, and member, of the Midlands Section Committee."
- 1948 G. G. Hopkinson (Gold Medal). "For Valuable Services rendered to the Society and to the Dyeing Industry."
- D. B. F. McAndrew (Silver Medal). "For Valuable Services rendered to the Society as Honorary Secretary and Committee Member of the Scottish Section 1935-1947."
- C. O. Clark (Silver Medal). "For Valuable Services rendered to the Society from 1923 to 1948."
- 1949 S. M. Neale (Gold Medal). "For his Pioneer Work in the Application of the Methods of Physical Chemistry to the Elucidation of the Phenomena of Dyeing, more particularly of Cellulosic Materials with Substantive Dyes."
- 1950 P. W. Cunliffe (Gold Medal). "For Outstanding Services to the Society and to the Tinctorial and Allied Industries."
- H. Foster (Gold Medal). "For Outstanding Services to the Society."
- L. A. Lantz (Bar attached to Silver Medal previously awarded). "For Exceptional Services to the Society and to the Tinctorial and Allied Industries in connection with Fastness Tests."

#### THE WORSHIPFUL COMPANY OF DYERS RESEARCH MEDAL

The Medal represents the Arms of the Worshipful Company of Dyers of the City of London, which were granted in 1471. The following is a brief description—

*Arms*—Sable, a chevron engrailed argent, between three bags of madder of the last, corded or.

*Crest*—On a wreath three sprigs of the graintree erect vert, fructed gules.

*Supporters*—Two leopards rampant gardant argent, spotted with various colours; fire issuing from their ears and mouth proper, both ducally crowned or.

*Motto*—*Da Gloriam Deo.*

1—The Dyers' Company offer annually a Gold Medal called "The Worshipful Company of Dyers Research Medal", the award of which is open to the Authors of papers embodying the results of scientific research or technical investigation connected with the tinctorial arts submitted to the Society of Dyers and Colourists, and published in the *Journal* of such Society during the twelve months ending on the 30th June in the year for which the Medal is granted, and, in the special circumstance provided for by Rule 6, during the twelve months ending on the 30th June in the year previous to that for which the Medal is granted. If a paper shall be published in two or more parts, then for the purpose of the award of the Medal, all the parts together shall be treated as a paper published in the year in which the final part is published.

2—The Medal will not be awarded to the same person on more than one occasion.

3—The Society of Dyers and Colourists shall consider the papers available for the Medal and advise the Company as to the merits thereof, and if, in the judgment of the Society, none of the papers is of sufficient merit, the Society may recommend that the Medal be not awarded.

4—The Dyers' Company will award the Medal either to the Author of the paper which, in all the circumstances, appears to the Company to show the greatest merit, or, in the event of such paper being the work of an Author who



has already been awarded the Medal, to the Author of the paper next in order of merit who has not already been awarded the Medal, and may, if the Company so thinks fit, refrain from making any award.

5—In the event of a paper being the work of two or more persons, the Author shall be taken to be that one of them whose work in the opinion of the Society of Dyers and Colourists has most substantially contributed to the merit of the scientific research or technical investigation embodied in such paper.

6—In the event of the Author of a paper of sufficient merit published in the *Journal of the Society of Dyers and Colourists* during any twelve months for which the Medal is awarded being unsuccessful in obtaining the award of the Medal for that period, the Society of Dyers and Colourists may, in their discretion, consider such paper for adjudication with the papers available for the award of the Medal for the next succeeding twelve months.

LIST OF RECIPIENTS

1908	Prof. E. Knecht. "A Means of Estimating the Degree of Mercerisation in Cotton Yarns."	1930-31	Prof. F. M. Rowe (Second Bar attached to Medal previously awarded), and Diplomas awarded to S. Ueno and F. H. Jowett as collaborators. "Insoluble Azo Colours on the Fibre and Action of Boiling Caustic Soda thereon."
1909	Prof. Arthur G. Green. "The Chemical Technology of Aniline Black."	1931-32	No award.
1910-11	R. L. Taylor. "The Action of Carbon Dioxide and of Air on Bleaching Powder."	1932-33	Two awards—W. T. Astbury. "The X-ray Interpretation of Fibre Structure." J. B. Speakman. "The Structure of the Wool Fibre; its Relation to the Dyeing and Finishing Processes of the Wool Textile Trade."
1911-12	W. Harrison. "The Electrical Theory of Dyeing."	1933-34	No award.
1912-13	S. H. Higgins. "Observations on the Bleaching of Cotton" and "The Action of Neutral Salts on Bleaching Solutions."	1934-35	H. A. Turner, and Diplomas awarded to G. M. Nabat and F. Scholefield, as co-authors. "The effect of Reduced Vat Dyes upon the Hypochlorite Oxidation of Cellulose."
1913-14	W. Johnson (in conjunction with Prof. Arthur G. Green, who had already been awarded the Medal). "The Constitution of Aged and of Bichromate Aniline Blacks."	1935-36	Prof. F. M. Rowe (Third Bar attached to Medal previously awarded), and Diplomas awarded to C. H. Giles, R. L. M. Allen, W. G. Dangerfield, and Glyn Owen, as collaborators. "Decomposition of Azo Dyes by Acids, Caustic Alkalies, and Reducing Agents."
1914-15	Morris Fort. "The Mechanism of the Acid Dye-bath."	1936-37	J. B. Speakman (Bar attached to Medal previously awarded), and Diplomas awarded to C. S. Whewell and J. L. Stoves, as collaborators. "The Reactivity of the Sulphur Linkage in Animal Fibres."
1915-16	James R. Hannay. "The Interaction between Metallic Copper and certain Dyes of the Thiazine, Oxazine and Azine Series."	1937-38	Prof. F. M. Rowe (Fourth Bar attached to Medal previously awarded), and J. B. Speakman (Second Bar attached to Medal previously awarded), and Diplomas awarded to E. Race and T. Vickerstaff, as collaborators. "The Uneven Dyeing of Wool with Acid and Chrome Dyes. Part I—The Reasons for the Uneven Dyeing Properties of Wool Staples and of Cloth Damaged by Exposure, and Part II—A Method for Correcting the Uneven Dyeing Properties of Wool Staples and of Cloth Damaged by Exposure."
1916-17	Prof. H. M. Dawson. "The Phenomena of Acid Catalysis and the Theory of Acids."	1938-39	No award.
1917-18	L. G. Radcliffe. "The Sulphonation of Fixed Oils."	1939-40	T. H. Morton, as senior author of the paper on "Application of Vat Dyes to Viscose Rayon", by J. Boulton and T. H. Morton.
1918-19	Chas. F. Cross (Diplomas presented to M. C. Lamb and C. V. Greenwood as co-authors). "Colloidal Tannin Compounds and their Applications."	1940-41	No award.
1919-20	A. E. Everest (Diploma presented to A. J. Hall as co-author). "The Tinctorial Properties of some Anthocyanins and certain Related Compounds."	1941-42	T. Vickerstaff, as senior author of the paper on "The Dyeing of Cellulose Acetate Rayon with Dispersed Dyes", by T. Vickerstaff and E. Waters.
1920-21	Prof. G. T. Morgan. "The Co-ordination Theory of Valency in Relation to Adjective Dyeing."	1942-43	No award.
1921-22	S. Judd Lewis. "On the Fluorescence of Cellulose and its Derivatives."	1943-44	J. Boulton. "The Importance of Dyeing Rate—an Interpretation for the Practical Dyer of Recent Research on Direct Dyeing."
1922-23	Prof. Arthur G. Green (Bar attached to Medal previously awarded), and K. H. Saunders. "The Ionamines—A New Class of Dyes for Cellulose Acetate Silk."	1944-45	No award.
1923-24	S. Judd Lewis (Bar attached to Medal previously awarded). "The Quantitative Determination of the Fluorescent Power of Cellulose and its Derivatives."	1945-46	No award.
1924-25	Prof. F. M. Rowe (with Diploma to Miss C. Levin, as co-author). "The Identification of Azo Colours on the Fibre and of Azo Pigments in Substance."	1946-47	H. Phillips, as senior author of the paper on "The Chemical Constitution and Physical Properties of Bisulphited Wool", by E. G. H. Carter, W. R. Middlebrook, and H. Phillips.
1925-26	H. H. Hodgson. "Behaviour of the Sulphides of Sodium in Aqueous and Alcoholic Media", and "The Action of Sulphur on the Monochlor-anilines."	1947-48	J. M. Preston. "Some Factors Affecting the Dyeing of Viscose" and "Some Aspects of the Drying and Heating of Textiles".
1926-27	Prof. F. M. Rowe (Bar attached to Medal previously awarded) and Diplomas awarded to collaborators—Miss E. Levin, A. C. Burns, J. S. H. Davies, and W. Tepper. "A New Reaction of Certain Diazosulphonates derived from $\beta$ -Naphthol-1-sulphonic acid, leading to the preparation of Phthalazine, Phthalazone and Phthalimidine Derivatives."	1948-49	H. Lindley, as major contributor to the paper "The Mechanism of Set and Supercontraction in Wool Fibres", by S. Blackburn and H. Lindley.
1927-28	No award.	1949-50	No award.
1928-29	F. Scholefield (with Diplomas to Miss E. Hibbert and C. K. Patel as co-authors). "The Action of Light on Dyed Colours."		
1929-30	H. H. Hodgson (Bar attached to Medal previously awarded). "Colour and Constitution from the Standpoint of Recent Electronic Theory."		

### THE WORSHIPFUL COMPANY OF FELTMAKERS RESEARCH MEDAL

The Feltmakers' Company offer annually a Gold Medal for Papers embodying the results of scientific research or technical investigation connected with the art of feltmaking and published in the *Journal* of the Society. Further, in order to encourage research of a type likely to qualify for the award of the Medal, the Feltmakers' Company offer annually, for an initial period of five years, a Research Grant of £50.

January 1945-June 1946 No award.

July 1948-June 1949 No award.

July 1946-June 1947 No award.

July 1949-June 1950 No award.

July 1947-June 1948 No award.

### KNECHT MEMORIAL FUND

A Fund of £100 was subscribed as a Memorial to the late Professor Edmund Knecht from the interest on which two prizes, in the form of books, are awarded annually to selected students of the Manchester College of Technology and the Royal Technical College, Salford.

### MERCER LECTURES

A sum of £20 per annum is being given to the Society for a limited period of years in commemoration of the centenary of the discovery of mercerization. Suitable lecturers are invited to give a Mercer Lecture annually.

1944	N. G. McCulloch and G. S. Hibbert.	"Science in an Old Industry."	1947	E. J. Bowen, M.A., F.R.S.	"Colour and Constitution—The Absorption of Light by Chemical Compounds."
1945	G. S. J. White, B.A., and T. Vickerstaff, Ph.D.	"Colour."	1948	F. Farrington, B.Sc., F.R.I.C.	"Textile Printing."
1946	D. Entwistle, B.Sc., A.R.I.C.	"Regenerated Fibres from Natural Polymers."	1949	E. Wilson, B.A.	"Some Applications of Chemistry to Textile Finishing."

### GEORGE DOUGLAS LECTURE

A sum of £2000 was given to the Society by the Bradford Dyers' Association Ltd., the income from the investment of which is employed in providing a biennial lecture on some subject connected with the colouring or finishing of textiles.

1949	H. Levinstein, M.Sc., Ph.D., M.I.Chem.E., F.R.I.C.	"George Douglas, His Times, and Some Thoughts on the Future."
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## Proceedings of the Society

### SCOTTISH AND NORTHERN IRELAND SECTIONS

Meetings held at the St. Enoch Hotel, Glasgow, on 29th November 1949, Mr. E. D. KENNEDY in the chair; and at the Queen's Hotel, Belfast, on 11th January 1950, Mr. J. H. JACKSON in the chair

### Dyeing Unions containing Nylon

J. G. GRUNDY

The outstanding physical and chemical properties of nylon have already ensured its place in the textile field, and as a result designers and manufacturers have explored the possibility of using this fibre in union with older textile materials. As the dyeing properties of nylon in many cases differ appreciably from those of other textile fibres with the same dyes, special attention has to be paid to the production of solid shades and two-coloured effects on unions containing nylon.

In this review the following unions have been considered—

- (1) Nylon and acetate rayon
- (2) Nylon and silk
- (3) Nylon and wool
- (4) Nylon and cellulosic fibres (cotton and regenerated cellulosic rayons).

#### (1) Nylon and Acetate Rayon

Dyes from the range of dispersed dyes for acetate rayon are suitable for dyeing solid shades on this union. Most dyes of this class dye the two fibres to a similar tone, when applied in the same dye-bath. Important exceptions occur in the oranges, scarlets, reds, and bordeaux. When dyes producing the above-mentioned hues are applied in combination with other dyes for the production of browns, the difference in tone produced by such dyes on the two fibres is fairly well masked, and in consequence there is no difficulty in producing shades commercially satisfactory in such tones as browns and greys. Therefore, the majority of shades which can be produced on acetate rayon with the dispersed class of dye may be equally well reproduced on a union of nylon and acetate rayon. Where there are exceptions to this rule, e.g. where shades of orange, scarlet, red, and bordeaux predominate, the shade on the nylon can be corrected to the acetate rayon by the judicious use of wool dyes, which do not dye acetate rayon but have an affinity for nylon in a neutral dye-bath.

It is possible to produce two-coloured effects on such a union. For this purpose the following points might be noted—

- (a) Some direct dyes will dye nylon and reserve acetate rayon.
  - (b) A large number of dyes from the various classes of water-soluble dyes used for wool also will dye nylon and reserve acetate rayon.
- Two-coloured effects can be produced by first dyeing the nylon with selected dyes of either group, and then overdyeing with dispersed dyes for acetate rayon, the ground shade already on the nylon being used as a basis for the two-coloured effects.

#### (2) Nylon and Silk

Unions of this type will most probably be confined to the hosiery trade, and the investigations to find suitable dyes for dyeing a union of these two fibres have been based mainly on the requirements of this branch of industry. It has been found that selected dyes from the range of dispersed dyes for acetate rayon, when applied at 95°C. without additions to the dye-bath, yield in many cases solid shades on the two fibres. Unfortunately, there are no blues which give a satisfactory depth on the pure silk, and, therefore, the usefulness of this range for this purpose is somewhat limited.

Much more satisfactory results can be obtained by selecting neutral-dyeing wool dyes, e.g. those of the Cloth Fast series. The following products, when applied with 10% Glauber's salt for 1 hr. at 95°C., yield full shades on the two fibres—

Cloth Fast Yellow 2RN  
Cloth Fast Brown RN  
Cloth Fast Black 4BN

With these three dyes most of the mode shades required for ladies' hose can be readily obtained.

Selected dyes from the Neolan range may also be used to produce solid shades on nylon and silk unions, and can be recommended where the highest standard of fastness is demanded. The dyes are applied with the addition of 3% formic acid for 1½ hr. at the boil.

Neolan Yellow GR  
Neolan Orange G  
Neolan Red REG  
Neolan Blue 2G  
Neolan Green BL conc.  
Neolan Black WA Extra

form a suitable basis for a wide range of shades. Neolan Black WA Extra is also suitable for producing blacks on the two fibres.

Carbide Black D, diazotized and developed with *m*-toluylenediamine, provides an alternative for a black shade.

#### (3) Nylon and Wool

Nylon unions of this class are being manufactured for most branches of industry. Such unions are proving of special interest for the production of light-weight materials for underwear and outerwear, and also for the manufacture of half-hose, etc. The problem of dyeing full shades on such unions has been investigated, and it is possible to make recommendations from most classes of wool dyes.

## (a) MOLECULARLY DISPERSED DYES

It has already been established that in the acid dye range monosulphonates have a greater affinity for nylon than disulphonates, and disulphonates a greater affinity than tri-sulphonates<sup>1</sup>. Unfortunately it is not possible to select dyes from the former class which are sufficiently compatible one with the other and sufficiently level-dyeing to dye wool in its commercial forms, e.g. yarn, hose, piece, and fabric. Such dyes would also be equally unsatisfactory for dyeing wool-nylon unions, for which it was therefore found necessary to make the selection from dyes having two sulpho groups, there being in this range a large number compatible one with the other, suitable for the production of level mode shades, and possessing the satisfactory fastness properties associated with goods of the above types. After some investigations, it was found that combinations of—

Kiton Fast Yellow 2G

Kiton Red G

Alizarine Sapphire Blue CB

gave reasonable results in medium depth over a wide range of tones, the only fault being the blue dye, which had insufficient affinity for the nylon as compared with the wool. The possibility of using a monosulphonated Alizarine Blue dye for this purpose was therefore investigated, the dye selected being Alizarine Sapphire Blue CR. With this dye it was found that the nylon had too great an affinity as compared with the wool. A compromise was then reached by using these blues in a 50:50 colour value proportion, and with the above red and yellow it was found possible to obtain a wide range of mode shades, in which the wool and nylon were dyed the same shade and depth.

These dyes are applied in accordance with the following instructions. Dyeing is carried out with the addition of—

10% Glauber's salt crystals

2% Sulphuric acid

The material is entered at 140°F. (60°C.), the temperature of the dyebath gradually raised to the boil, and boiling continued for 45–60 min.

These dyes are suitable for producing solid shades on the two fibres only in medium depths. In light shades the nylon is dyed heavier than the wool, whilst in heavy shades the wool is dyed heavier than the nylon.

The behaviour of these dyes on the two fibres dyed in separate baths and on a 50:50 wool-nylon union is shown in Fig. 1–12. These graphs were obtained by carrying out dyeings at various temperatures lower than the boil, and making a visual estimate of the strength of the resulting dyeing by comparing it with a set of standard dyeings made at 100°C. It follows, therefore, that all the dyeings on separate fibres show an apparent exhaustion of 100% at 100°C. The temperature of the dyeing is plotted along the horizontal axis and the apparent depth of shade produced at any temperature is plotted along the vertical axis.

The soundness of the procedure was established by the results of colorimetric estimations for the dyes in question. It was noted, by this procedure, that the exhaustions on the wool and nylon were

fairly similar for all practical purposes and for the depths of shade reviewed. The exhaustion was over 80% in each case.

In Fig. 1–3 the results of carrying out such a temperature range investigation are shown for 0.1% shades of Kiton Fast Yellow 2G, Kiton Red G, and the mixture of Alizarine Sapphire Blues referred to above, on the individual fibres in separate baths. The apparent depth produced is greater the higher the temperature at which the dyeing has been made, and is, of course, for the reason explained above, 0.1% at the boil. The graphs for each dye are of the same general type, and show the apparent depth produced to be somewhat greater for wool than for nylon at all temperatures below the boil.

In Fig. 4–6 the same procedure has been carried out, with the same three dyes at the same depth, on a 50:50 wool-nylon union. The three dyes again show the same general behaviour. There is a dyeing temperature (about 70°C.) at which a solid shade may be produced; but at the practical dyeing temperature of 100°C. or just under, the nylon is dyed much heavier than the wool.

In Fig. 7–9 the same dyes have been used but in a 1% depth on the separate fibres. It will be noted that, as was the case in dyeing pale shades with these dyes, the curves for nylon at the medium depths are closely similar to those for wool.

In Fig. 10–12 a 1% shade of the same three dyes has been dyed on a 50:50 wool-nylon union. It will be seen that at 100°C. the 1% depth of shade yields a more nearly solid union dyeing than did the 0.1% dyeing. It is thus clearly shown that, when using dyes of the molecularly dispersed class, the two fibres are dyed to similar depths at medium shades, but the nylon is dyed very much heavier than the wool in light shades; these dyes are, therefore, suitable only under normal dyeing conditions for obtaining solid effects on wool-nylon unions in medium depths and should not be used for pale shades (but cf. p. 11). This difference between pale and heavy shades is not so marked with the Alizarine Sapphire Blue mixture as with the other two dyes under investigation, owing to the presence in the blue mixture of both a mono- and a di-sulphonic acid.

Rate-of-dyeing curves for Kiton Fast Yellow 2G, at 0.1%, 1.0%, and 5% depths, have been determined, and are shown in Fig. 13–15.

When dyed in 0.1% depths in separate baths (Fig. 13) the wool speedily reaches a “visual” equilibrium, whereas the nylon continues to lose in depth, owing to continuing penetration of the fibre by the dye. When the two fibres are both present in the one bath, this penetration of the nylon fibre is almost exactly counterbalanced by dye boiling off the wool and on to the nylon, thus causing the wool to lose in depth whilst maintaining the depth on the nylon. This is to be expected, since nylon has a greater avidity for dye than wool, and at a 0.1% shade the saturation value of the nylon has yet to be reached.

The same dye, when used at 1% concentration (Fig. 14), has approximately the same behaviour on the two fibres separately and in the form of a

## Temperature Range of 0.1% Shades of Molecularly Dispersed Dyes on Wool and Nylon

(FIG. 1-6)

## Separate Baths

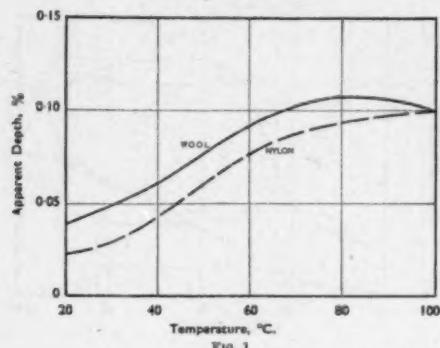
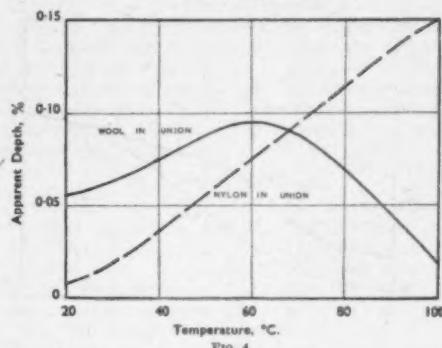


FIG. 1

## Union Materials



Kilon Fast Yellow 2G

FIG. 4

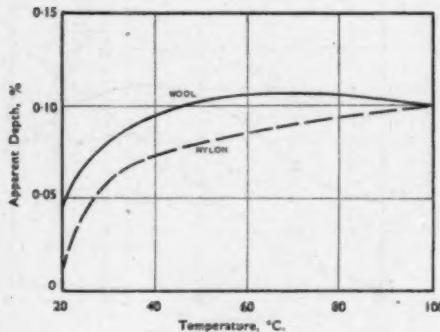


FIG. 2

Kilon Red G

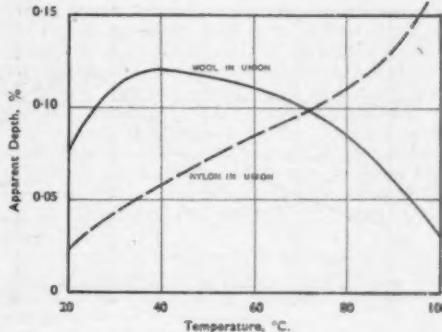


FIG. 5

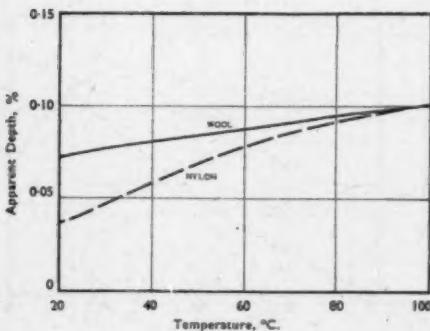


FIG. 3

50:50 Alizarine Sapphire Blue CB + Alizarine Sapphire Blue CR

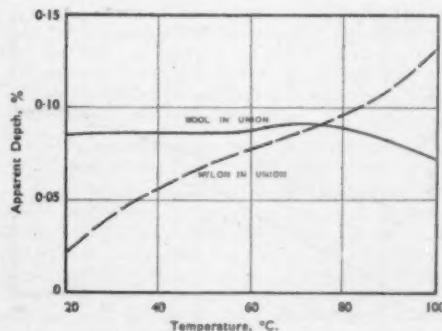


FIG. 6

**Temperature Range of 1% Shades of Molecularly Dispersed Dyes on Wool and Nylon**  
(FIG. 7-12)

**Separate Baths**

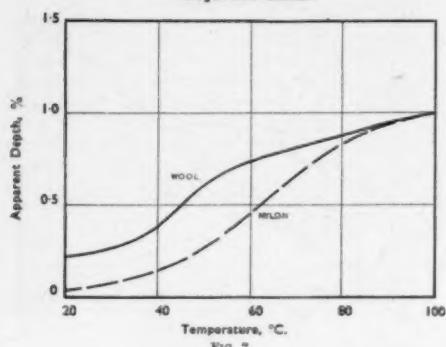


FIG. 7

Kiton Fast Yellow 2G

**Union Materials**

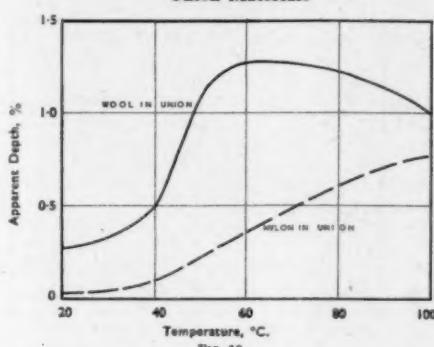


FIG. 10

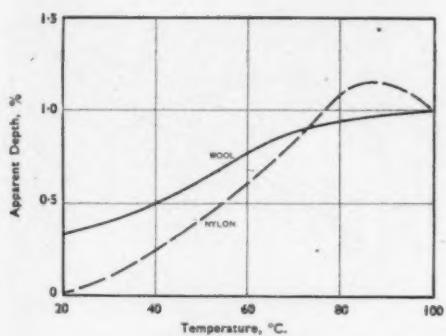


FIG. 8

Kiton Red G

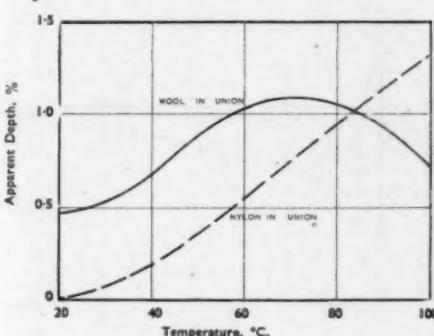


FIG. 11

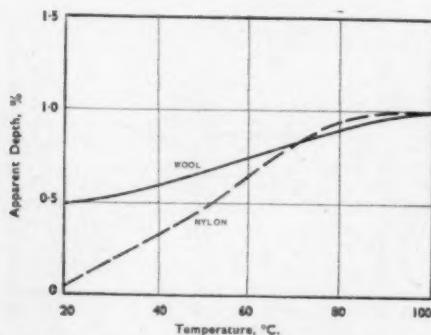


FIG. 9

50:50 Alizarine Sapphire Blue CB + Alizarine Sapphire Blue CR

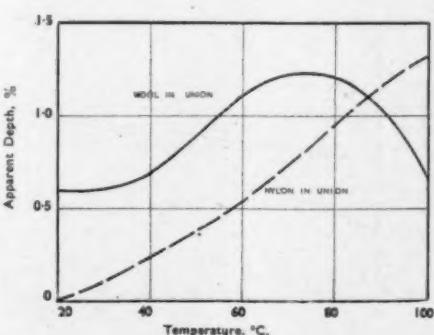


FIG. 12

**Rate of Dyeing of Kiton Fast Yellow 2G on Wool and Nylon at 100°C.**

(FIG. 13-15)

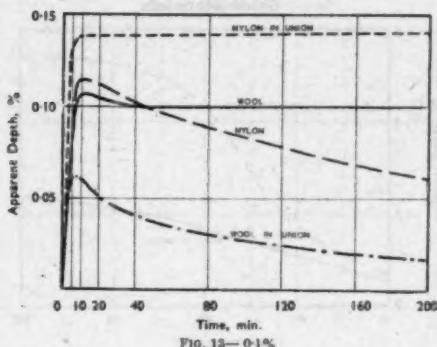


FIG. 13—0.1%

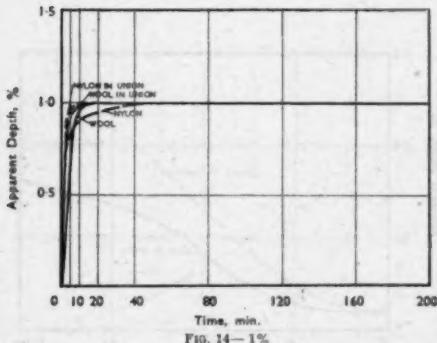


FIG. 14—1%

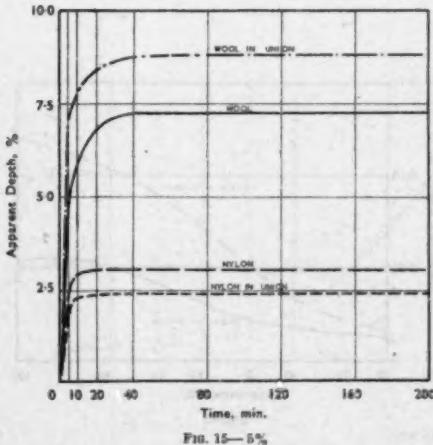


FIG. 15—5%

union, and solid shades are obtainable at this depth.

When a 5% concentration of the same dye is applied (Fig. 15), the saturation value of the nylon is always reached whether present in union or otherwise, and approximately the same depth of shade on the nylon is always obtained. In this concentration, when dyeing a union of the two fibres, the wool absorbs about four times as much dye as the nylon. Solid shades are not therefore obtainable in dyebaths of high concentration, with dyes of this class.

**(b) AGGREGATED DYES**

With this class of dye there is no theoretical indication as to the suitability of the products either for dyeing nylon or for dyeing a union of wool and nylon. In consequence, the dyes suitable for the latter purpose have had to be discovered by carrying out practical trials.

The knowledge gained on the application of these dyes to all-wool material was, of course, made use of. As it is well known that dyes of this class are not very suitable for dyeing all-wool materials in combination shades, particularly for the lighter tones, it was assumed that the application of these dyes would be equally difficult for such tones when dyeing a wool-nylon union, on account of the poor levelling properties of such dyes. It was therefore decided to carry out investigations with a view to determining dyes suitable for producing full shades of yellow, brown, scarlet, and red, and also maroons, navy blues, dark greens, and blacks.

In general, the dyes are applied from a neutral bath, entering at 50–60°C., raising the temperature to the boil, and boiling for 45–60 min. In one or two instances the process is modified by adding 5% ammonium acetate and 10% Glauber's salt to the dyebath.

The dyeing properties of the selected Cloth Fast and other similar wool dyes are indicated in Fig. 16–27, obtained in the same manner as the previous graphs.

Fig. 16–19 show the dyeing properties of Cloth Fast Yellow 2RN in 1% and 3% shades on a wool-nylon union and on the fibres dyed separately. It will be noted that in the 1% shades the nylon in the union is dyed much heavier than the wool, but with increasing depth this difference is reduced, thereby indicating the special suitability of these dyes for producing solidity on the two fibres in full shades.

These comments are further confirmed by Fig. 20–23 illustrating Wool Red 3G and Fig. 24–27 illustrating Cloth Fast Blue RN.

**PALE SHADES DYED WITH MOLECULARLY  
DISPERSED AND AGGREGATED DYES**

As previously explained, when pale shades are dyed with level-dyeing acid dyes and neutral-dyeing dyes of the Cloth Fast class, the nylon is dyed much heavier than the wool. The amount of dye taken up by the nylon can be controlled by the addition of Claytavon E or other anionic detergent to the dyebath. A concentration of 0.5–2 c.c. per litre is required according to the dye and the blend of nylon and wool. It is possible by means of such

## Temperature Range of Aggregated Dyes on Wool and Nylon

(Fig. 16-21)

## Separate Baths

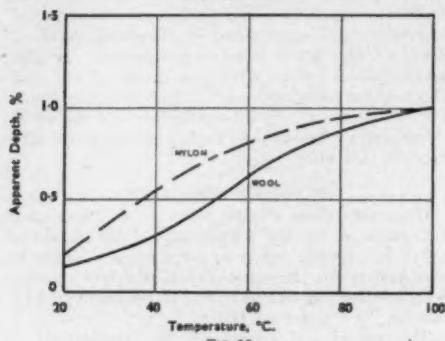


FIG. 16

## Union Materials

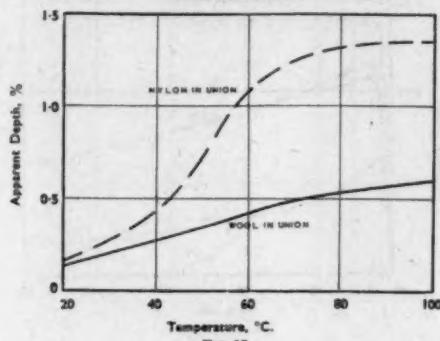


FIG. 17

1% Cloth Fast Yellow 2RN

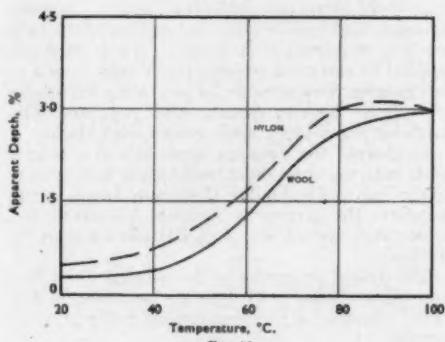


FIG. 18

3% Cloth Fast Yellow 2RN

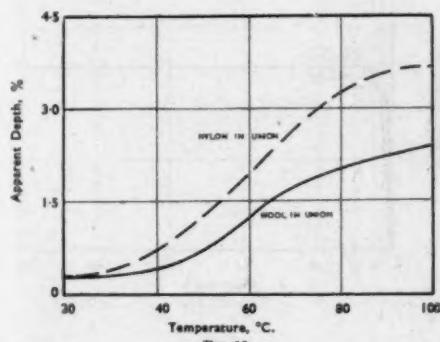


FIG. 19

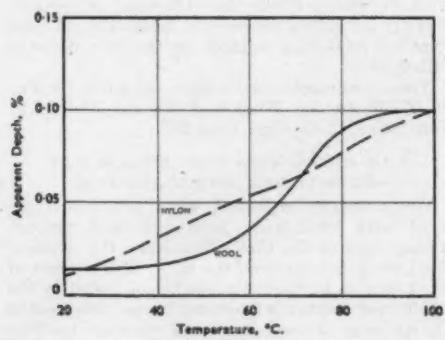


FIG. 20

1% Wool Red 3G

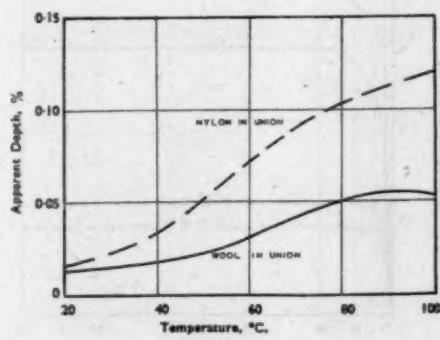


FIG. 21

## Temperature Range of Aggregated Dyes on Wool and Nylon

(FIG. 22-27)

## Separate Baths

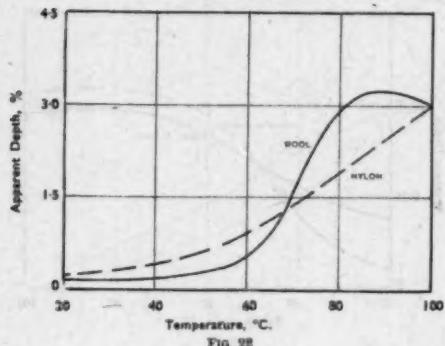


FIG. 22

2% Wool Red 90

## Union Materials

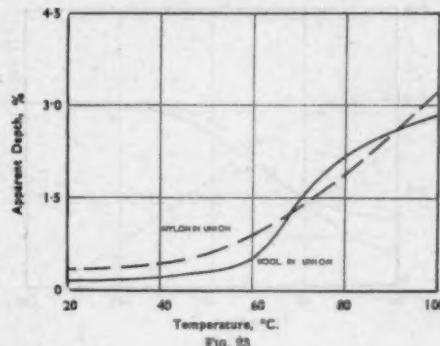


FIG. 23

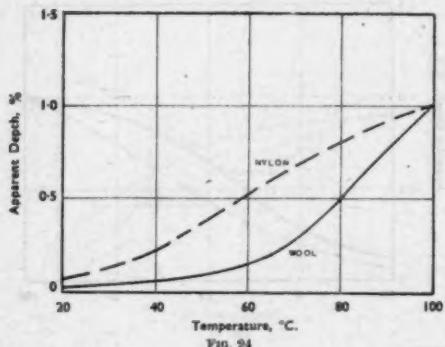


FIG. 24

1% Cloth Fast Blue RN

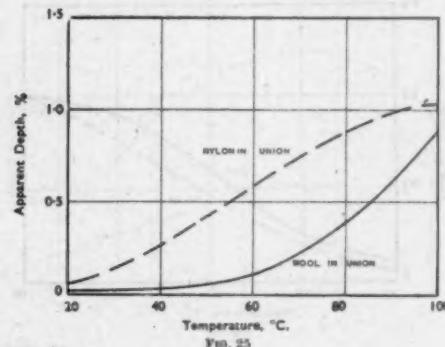


FIG. 25

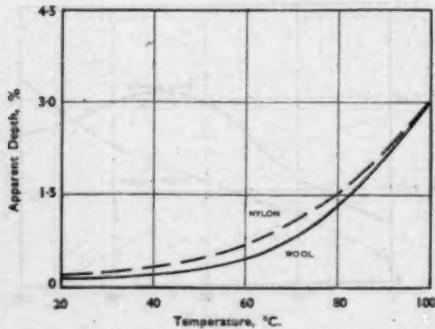


FIG. 26

2% Wool Red 90

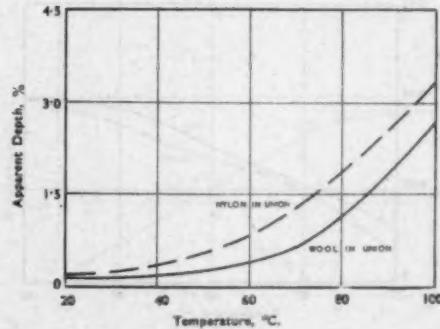


FIG. 27

2% Wool Red 90

## Temperature Range of Premetallized Dyes on Wool and Nylon

(FIG. 28-33)

## Separate Baths

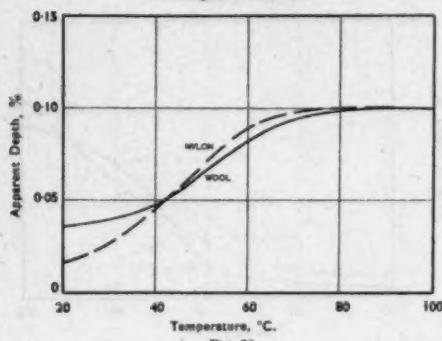


FIG. 28

0.1% Neolan Orange G

## Union Materials

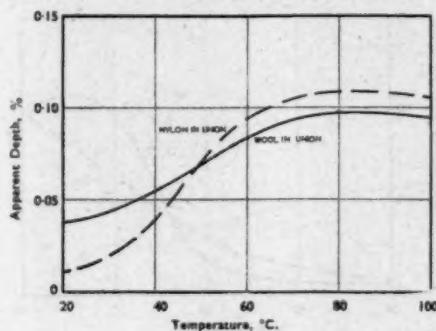


FIG. 29

0.1% Neolan Orange G

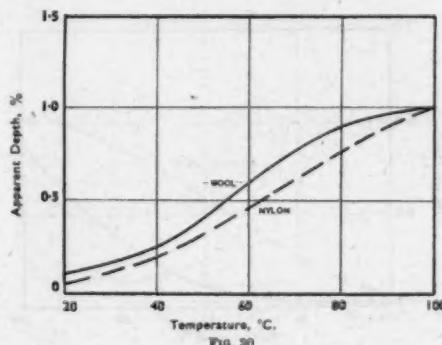


FIG. 30

1% Neolan Orange G

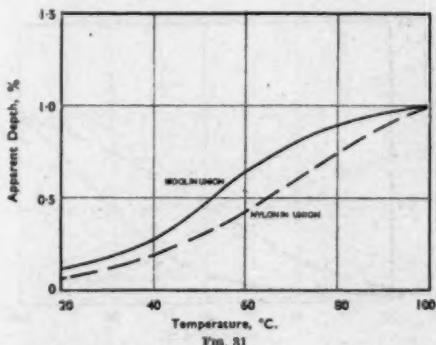


FIG. 31

1% Neolan Orange G

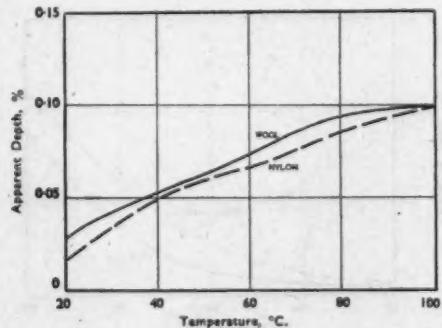


FIG. 32

0.1% Neolyn Red BEG

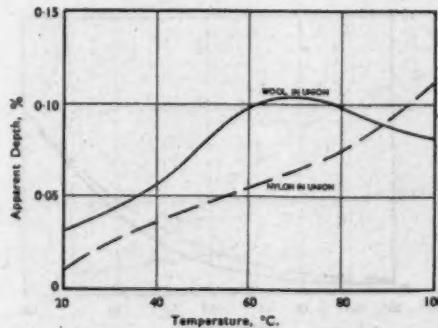


FIG. 33

## Temperature Range of Premetallized Dyes on Wool and Nylon

(FIG. 34-39)

## Separate Baths

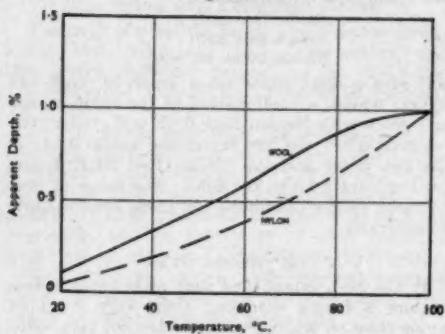


FIG. 34

## Union Materials

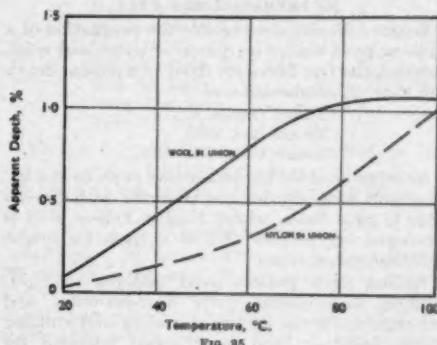


FIG. 35

1% Neolan Red RRG

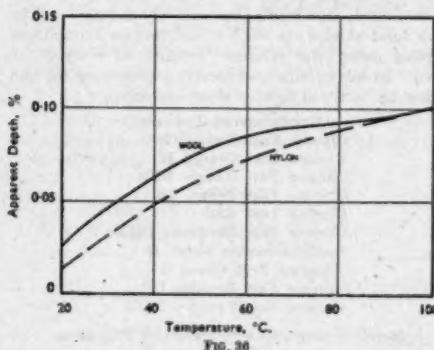


FIG. 36

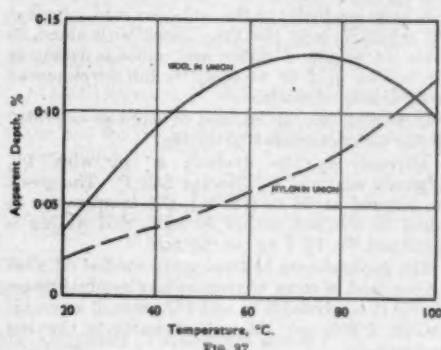


FIG. 37

0.1% Neolan Green BL conc.

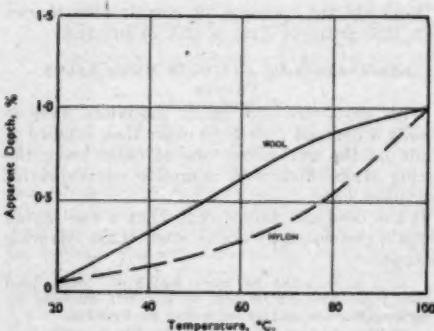


FIG. 38

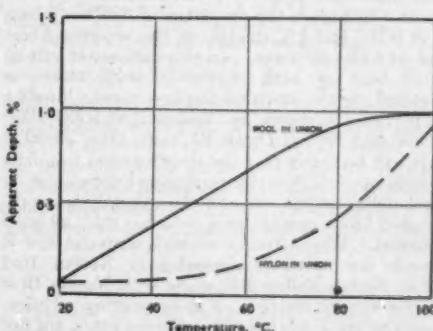


FIG. 39

1% Neolan Green BL conc.

additions to the dyebath to obtain solidity on the two fibres when dyed in pale shades, with these groups of dyes.

(c) PREMETALLIZED DYES

Selected Neolan dyes enable the production of a wide range of shades on unions of nylon and wool, in which the two fibres are dyed to a similar depth and tone. A combination of—

Neolan Orange G  
Neolan Red REG  
Neolan Green BL conc.

is recommended for the production of mode shades. Blue and navy shades are obtained with Neolan Blue G as a basis, whilst Neolan Yellow 8GE is employed for yellows and as a basis for bright yellowish-green tones.

Neolan dyes possess good fastness to light, washing, perspiration, water, and sea-water, and are suitable for the dyeing of hosiery and knitting yarns, half-hose, and piece goods intended for light-weight tropical suiting and ladies' blouse and dress materials.

The dyebath is prepared with 4% sulphuric acid 168°Tw. and the material entered after previous wetting out. Run for 10 min., add the dissolved dye, raise gradually to the boil, then add a further 4% sulphuric acid 168°Tw. diluted with about 20 times its weight of water, and continue dyeing at the boil for 1½–2 hr. to complete full development and levelling of shade.

If desired, the full amount of acid can be added at the commencement of dyeing.

Alternatively the dyebath is set with 4% sulphuric acid and 4% Neolan Salt P. The goods are entered at 50°C. (120°F.), the temperature is raised to the boil within 30 min., and dyeing is continued for 1½–2 hr. at the boil.

The goods should be thoroughly washed off after dyeing, and in order to remove any residual traces of acid it is advisable to add 1–2 pints of ammonia (sp. gr. 0.880) per 100 gal. of water to the last rinsing bath.

Neolan dyes offer an advantage over the molecularly dispersed and aggregated dyes, inasmuch as they are suitable for dyeing solid shades on wool-nylon unions in both pale and medium shades. This important property is demonstrated by Fig. 28–31, which show the properties of Neolan Orange G in 0.1% and 1% shades, on the separate fibres and on a 50:50 union. In this instance it will be noted that at both depths a solid shade is obtained on the union of the two fibres. Similar properties are shown by Neolan Red REG (Fig. 32–35) and Neolan Green BL conc. (Fig. 36–39).

It will be noted that the dyes selected from the Neolan range of dyes, for producing solid shades on wool-nylon unions, are not the usual dyes recommended for combination shades on all-wool material. When dyeing all-wool material the E brands are normally applied, e.g. Neolan Red BRE, Neolan Yellow BE, along with Neolan Blue 2G for the production of mode shades. Unfortunately, the E brands of the Neolan range are not very suitable for dyeing nylon, and hence the rather unusual recommendation had to be made for

dyeing nylon unions. However, use can be made of the lack of affinity of the E brands on nylon to produce two-coloured effects on wool-nylon unions. For example a combination—

Neolan Orange G  
Neolan Red REG  
Neolan Green BL conc.

will give a solid shade on a union of wool and nylon; whilst a combination of the same orange and green with Neolan Red BRE will give a two-coloured effect, the first two giving a solid shade on the two fibres and the Neolan Red BRE dyeing predominantly on to the wool. The range of two-coloured effects obtainable on these lines is quite considerable.

(d) CHROME DYES

It has been established that most chrome dyes require a longer chroming time when dyed on nylon than on wool. Such dyes are not very satisfactory for application to light-weight wool-nylon unions, since to bring both fibres to the same shade a prolonged chroming time is required, which is reflected in the quality of the finished goods.

The usefulness of this range of dyes for dyeing wool-nylon unions is somewhat limited. It has been found, however, that the dyes listed below give solid shades on wool-nylon unions in medium depths using the normal method of dyeing for wool. In heavy shades there is a tendency for the nylon to be dyed lighter than the wool.

AFTERCROME PROCESS  
Chrome Fast Yellow G  
Chrome Fast Orange R  
Chrome Fast Orange M2R  
Chrome Fast Brown 2R  
Chrome Fast Red G  
Chrome Fast Bordeaux BRN  
Naphthochrome Violet R  
Alizarine Fast Green G  
Chrome Fast Cyanine GP  
Chrome Fast Cyanine 2BSS

METACHROME AND SYNCHROME PROCESSES

Chrome Fast Yellow G  
Chrome Fast Brown 2R  
Synchromate Brown RLL  
Chrome Fast Red G  
Synchromate Red GR  
Alizarine Fast Green G

Work on the question of combination shades with this group of dyes is still in progress.

RESERVATION OF NYLON IN WOOL-NYLON UNIONS

With most two-fibre union materials there is usually a demand for effects other than solidity of shade on the two fibres, one of these being the dyeing of one fibre with complete reservation of the other fibre.

It has been ascertained that when a wool-nylon union is pretreated by one or other of the following methods—

(i) 5 g. Invadine BL conc. and 3 c.c. formic acid (80%) per litre for 30 min. at the boil, followed by hydroextraction and entering into the dyebath.

(ii) 5 g. Albatax WSE per litre for 30 min. at the boil, followed by hydroextraction and entering into the dyebath.

almost complete reservation of the nylon is obtained with a wide range of acid, Cloth Fast, Neolan, and chrome dyes.

#### (4) Nylon and Cellulosic Fibres

A variety of possibilities are available for dyeing nylon and cellulosic fibres (e.g. cotton and regenerated cellulosic rayons) in the one bath. Taken in order of simplicity, they may be described as follows—

(a) Dyeing the nylon and the cellulosic fibre in the same, mildly alkaline bath with a combination of dispersed acetate rayon dyes and selected direct dyes which have little or no affinity for nylon. In general, dyeings produced by this technique are reasonably fast to light over a wide range of hues, but of only moderate fastness to washing.

(b) Dyeing the nylon with selected neutral-dyeing wool dyes and the cellulosic fibre with selected direct dyes in the same bath.

(c) Dyeing the nylon first with selected wool dyes having little or no affinity for cellulosic fibres, from an acid bath, and afterwards filling in the cellulosic fibre in a second bath with selected direct dyes.

(d) Dyeing the cellulosic fibre with selected vat dyes which have little or no affinity for nylon, and cross-dyeing the nylon with—

- (i) Selected dispersed dyes for acetate rayon
- (ii) Selected water-soluble wool dyes from the various ranges available for this purpose.

The following vat dyes applied on the jigger by the normal process for cellulosic fibres possess very little affinity for the nylon fibre—

CIBANONE

Yellow GN  
Golden Orange 3G, 2R

Brilliant Orange GK, RK  
Brilliant Pink 2R  
Red 4B, FBB, 2B  
Brown GR, BG, BR, 2BR, RV, 3B  
Red Brown R  
Brilliant Violet ER  
Olive 2R, 2B, BG  
Brilliant Green EBF, E2G  
Blue GF, RS  
Navy Blue ERA  
Grey BG, 2GR

All the combinations of dyes referred to are equally suitable for the production of two-coloured effects on unions of nylon with cellulosic fibres.

#### Conclusions

I—Nylon in union with (1) acetate rayon, (2) silk, (3) wool, and (4) cellulosic fibres (cotton and regenerated cellulosic rayons) can be dyed to solid shades possessing varying degrees of fastness to meet the requirements of most of the industries associated with these fibres.

II—The unions described under I can be dyed to a limited extent in two-coloured effects, by means of one- and two-bath processes.

III—The effects described under I and II can be obtained by means of the judicious choice of dyes and by the normal dyeing techniques for the above fibres.

I must express my indebtedness to the Clayton Aniline Co. Ltd. for permission to publish this paper and for the assistance of the dyehouse staff in compiling it.

#### Reference

<sup>1</sup> Abbot, Crook, and Townend, J.S.D.C., 63, 462 (Dec. 1947).

### COMMUNICATIONS

#### The Reaction of Wool with Organic Chloroamines

#### II—The Nature of the Reaction

P. ALEXANDER, D. CARTER, and C. EARLAND

It is shown that chloroamines can oxidize wool by two different mechanisms, although in neither case does reaction occur via the intermediary of free chlorine in solution. Only one of the reactions renders wool non-felting, and it is favoured by the presence of chloride ions in solution and by increasing the electronegativity of the group adjacent to the reactive  $>\text{NCl}$  group. Amino acid analyses of treated wool and reaction with model substances show that the disulphide bond is equally readily oxidized by both mechanisms, although they differ in their reaction with tyrosine, which, it is suggested, must be oxidized for the production of non-felting wool by surface degradation.

#### Introduction

In Part I of this investigation<sup>1</sup> it was shown that chloroamines in solution below pH 2 reacted rapidly with wool, but that resistance to felting was obtained only with two or three times as much active chlorine as was necessary when chlorine itself was used in acid solution. When, however, the reaction was performed in the presence of high concentrations of chloride ions, certain chloroamines gave a completely non-felting wool with low quantities of active chlorine. It was shown that chloride ions were specific for this purpose and that the different chloroamines varied in reactivity.

Thus, chlorosulphamic acid was an excellent anti-felting agent, while alkylchloroamines were almost ineffective. The object of this paper is to determine the influence of the structure of the chloroamine and the effect of chloride ions on the action of these compounds and to determine which groups in the wool have to be oxidized for non-felting to result.

#### Experimental

##### 1. REACTION KINETICS

Patterns of wool fabric were treated with a solution of the chloroamine using the procedure described in Part I. The course of the reaction was

followed by removing aliquot portions of the solution at definite intervals of time and titrating iodometrically with 0.10 N. sodium thiosulphate solution.

## 2. EXPERIMENTS ON MODEL SUBSTANCES

The rates of reaction between *N*-monochlorourea or *N*-chlorosulphamic acid and hydrolytic degradation products of wool keratin, i.e.  $\alpha$ -amino acids, and substances related to keratin, e.g. silk, polyamino acids, and nylon, were determined using the procedure described under §1. 0.4 g. of the substance, or 5 g. in the case of silk or nylon, was stirred at 40°C. with 400 ml. of 0.50% sulphuric acid containing the chloroamine, equivalent to 0.25 g. of available chlorine. The half-life of the reaction was determined in the absence of chloride ions and with 10.0% sodium chloride in solution. All the chemicals employed were obtained from laboratory suppliers and used without purification. Polyglycine and polyalanine, each containing approximately twenty amino acid residues, were obtained by condensing the *N*-carboxyanhydrides of the respective amino acids<sup>2</sup>.

## 3. CHEMICALLY MODIFIED WOOL

Wool fabric was acetylated with acetic anhydride and sulphuric acid<sup>3</sup>. The reduced acid-combining power showed that 77% of the basic groups had been acetylated. The carboxyl groups were methylated with a solution of diazomethane in ether<sup>4</sup>. Some of the disulphide bonds were converted to lanthionine (a) with a large excess of 0.10 N. sodium hydroxide solution for 24 hr. and (b) by boiling in a pH 8 buffer solution for 2 hr.<sup>5</sup>

## 4. CYSTINE ANALYSES

The wool sample was hydrolysed in 5 N. hydrochloric acid at 120°C. for 5 hr. in a sealed tube. The cystine content of the hydrolysate was determined colorimetrically with phosphotungstic acid using a Spekker photoelectric absorptiometer<sup>6</sup>.

## 5. TYROSINE ANALYSES

A 0.5-g. sample of the wool was hydrolysed in 5 N. sulphuric acid at 120°C. for 5 hr. in a sealed tube. The tyrosine was estimated using an adaptation due to Lugg<sup>7</sup> of the Folin-Cioateau colorimetric method based on the Millon reaction.

## 6. SUPERCONTRACTION MEASUREMENTS

One-gram bundles of Lincoln fibres were wetted out, and the distance between two tie bands was measured with a ruler. After treatment the fibres were withdrawn and this distance was again measured. The supercontraction is expressed as the percentage reduction in length.

## 7. DETERMINATION OF FREE CHLORINE IN THE PRESENCE OF CHLOROAMINES

The free chlorine in solutions containing chloroamines was determined in two ways—(a) by aspirating nitrogen through the solution and determining the concentration of chlorine in the gas stream by passing this through acidified potassium iodide solution, and (b) by extracting with carbon tetrachloride. The rate of bubbling through, or the time of contact of the carbon tetrachloride with,

the solution of the chloroamine was adjusted to ensure that equilibrium was set up between the two phases.

Some samples of carbon tetrachloride contained impurities which reacted with chlorine. These were removed by chlorinating the solvent, and after standing for several days removing the excess of chlorine by shaking with mercury and finally distilling.

These methods are obviously unsuitable for estimating chlorine in the presence of volatile chloroamines, e.g. *N*-chloromonoethylamine, or chloroamines such as *NN*-dichloro-*p*-toluenesulphonamide which are soluble in carbon tetrachloride. *N*-Monochlorourea and *N*-chlorosulphamic acid are, however, non-volatile and insoluble in carbon tetrachloride, and the concentration of free chlorine in these solutions could thus be determined knowing the partition coefficient of chlorine between water and nitrogen or carbon tetrachloride. This partition coefficient of chlorine between the two phases was determined by bubbling nitrogen through, or by extracting with carbon tetrachloride, a dilute solution of chlorine in water, adjusted to pH 1.5 with sulphuric acid, in both the absence and the presence of 10.0% sodium chloride in the solution.

## Results

It has been generally accepted that the reaction of chloroamines is always preceded by their hydrolysis and that the reacting species is in fact chlorine in solution (see “Discussion”, p. 21). To examine this probability the concentration of chlorine in equilibrium with chloroamines was examined.

### 1. THE FREE CHLORINE IN AQUEOUS SOLUTIONS OF *N*-MONOCHLOROUREA AND CHLOROSULPHAMIC ACID

To determine if the rôle of the chloride ions was to displace the following equilibrium to the right (i.e. to produce more free chlorine)—

$$R-NHCl + H^+ + Cl^- \rightleftharpoons R-NH_2 + Cl_2$$

solutions of *N*-monochlorourea and chlorosulphamic acid in 0.50% sulphuric acid solution, of the same concentrations as were used for treating wool, were analysed for free chlorine in the absence and presence of 10.0% sodium chloride.

(a) PARTITION METHOD — The partition coefficient of chlorine between carbon tetrachloride and 0.50% sulphuric acid solution at 40°C. was determined in the presence and absence of sodium chloride in the solution (Table I).

TABLE I  
Partition of Chlorine between Carbon Tetrachloride and 0.50% Sulphuric Acid

Electrolyte	Vol. of 0.10 N- $Na_2S_2O_3$	Partition Coefficient	
	= 25 ml.	= 5 ml.	
	aqueous	$CCl_4$	
	soln.	soln.	
None	12.33	9.80	4.0
2.0% NaCl	2.14	9.86	23.9
5.0% NaCl	1.89	9.10	24.1
10.0% NaCl	1.67	8.43	25.2
2.0% $NaNO_3$	10.83	8.09	3.7
10.0% $NaNO_3$	9.40	8.20	4.4

The addition of sodium chloride thus increased the partition coefficient approximately sixfold. This was not due to a “salting out” effect, as the partition coefficient was not increased by the addition of sodium nitrate.

The experiment was then repeated by substituting for the chlorine solution a solution of *N*-monochlorourea in 0.50% sulphuric acid of the same concentration as was used for treating the wool.

From Table II it is seen that the concentration of free chlorine was not increased by the addition of sodium chloride.

TABLE II  
Concentration of Free Chlorine in  
*N*-Monochlorourea Solution

NaCl in soln. (%)	Vol. of 0.01 n- $\text{Na}_2\text{S}_2\text{O}_3$ 25 ml. $\text{CCl}_4$ soln. (ml.)	Partition Coefficient	Concn. of $\text{Cl}_2$ in $\text{CCl}_4$ layer (g.-mol./ litre)	Concn. of $\text{Cl}_2$ in <i>N</i> -Monochloro- urea soln. (g.-mol./ litre)
0	0.15	4	$3 \times 10^{-8}$	$7.5 \times 10^{-8}$
10	0.50	24	$10 \times 10^{-8}$	$4 \times 10^{-8}$

(b) ASPIRATION METHOD—When nitrogen was passed through dilute acid chlorine solutions the addition of 10.0% sodium chloride approximately doubled the concentration of chlorine in the gaseous phase. When the chlorine solution was replaced by a solution of *N*-monochlorourea or chlorosulphamic acid of pH 1.5 the addition of sodium chloride also doubled the chlorine in the nitrogen, i.e. the actual concentration of chlorine in the chloroamine solution was unchanged by the addition of chloride ions.

These experiments established that the concentration of chlorine in equilibrium with a chloroamine in acid solution is very small and is not increased by the addition of sodium chloride. However, the thermodynamic activity of the chlorine is greatly increased in the presence of chloride ions, and it was thought possible that this increase in activity of the small quantity of chlorine in equilibrium with the chloroamine brought about by chloride ions might account for the enhanced anti-felting effect. This explanation cannot be correct, however, since the addition of sodium chloride to an aqueous solution of chlorine at pH 1.5 does not render the solution more effective for making wool unshrinkable (Table III).

TABLE III

Addition of Sodium Chloride to Chlorine Solutions					
Felting Procedure	Area Untreated	Shrinkage (%) of Pattern 1.5% $\text{Cl}_2$ no NaCl	1.5% $\text{Cl}_2$ in 20% NaCl	1.5% $\text{Cl}_2$ no NaCl	1.5% $\text{Cl}_2$ no NaCl
Washing machine for 53 min. ... ...	25.4	7.9	8.8		
Washing machine for 106 min. ... ...	33.0	14.3	14.6		
Milling stocks for 30 min. ...	32.1	14.6	14.5		
Milling stocks for 60 min. ...	42.6	30.4	26.4		

## 2. KINETICS OF THE REACTION

The kinetics of the reaction between chlorine and wool has been extensively studied<sup>8-10</sup>, and it was

immediately obvious that the rate of reaction of chloroamines (see Part I<sup>1</sup>) was much too great to be explained in terms of the very low chlorine concentration found in equilibrium. Other aspects of the kinetics will also be seen to exclude the possibility that the reaction proceeds via free chlorine in solution.

It was found that the reaction between chloroamines and wool was always of the first order with respect to chloroamine concentration, i.e. the rate constant *k* was given by—

$$k = \frac{1}{t} \ln \frac{a}{a-x}$$

where *a* = initial concentration of chloroamine and *x* = amount of chloroamine changed in time *t*. The rate constants for typical treatments of wool with *N*-monochlorourea and *N*-chlorosulphamic acid are given in Tables IV and V respectively.

TABLE IV

### Reaction between Wool and *N*-Monochlorourea

(*N*-Monochlorourea (equivalent to 1.8%  $\text{Cl}_2$ ) in 10.0% NaCl solution of pH 1.5 at 40°C.)

Time <i>t</i> (min.)	Vol. <i>V</i> of 0.10 n- $\text{Na}_2\text{S}_2\text{O}_3$ = 25 ml. of bath	$k = \frac{2.3}{t} \log \frac{V_0}{V_t}$ (ml.)
0	1.59	—
10	0.92	0.055
20	0.50	0.058
30	0.30	0.056
40	0.21	0.051
50	0.15	0.047
60	0.08	0.030

TABLE V

### Reaction between Wool and *N*-Chlorosulphamic Acid

(*N*-Chlorosulphamic acid (equivalent to 4.0%  $\text{Cl}_2$ ) in 10.0% NaCl and 0.50%  $\text{H}_2\text{SO}_4$  soln. at 40°C.)

Time <i>t</i> (min.)	Vol. <i>V</i> of 0.10 n- $\text{Na}_2\text{S}_2\text{O}_3$ = 10 ml. of bath	$k = \frac{2.3}{t} \log \frac{V_0}{V_t}$
0	1.35	—
5	0.77	0.112
10	0.54	0.091
15	0.40	0.081
20	0.28	0.079
25	0.18	0.081

It was shown in Part I<sup>1</sup> that the half-life of the reaction using *N*-chlorosulphamic acid is independent of the chloride ion concentration, but with *N*-monochlorourea the half-life decreases with increasing chloride ion concentration. Therefore, only when wool is treated with *N*-monochlorourea is the rate constant affected by the addition of soluble chlorides.

The apparent activation energy of the chlorosulphamic acid process was calculated from the velocity constants determined at two different temperatures and was found to be 12.7 kg.cal. per mole. In Part I it was shown that the rate of reaction was independent of stirring in the region studied, and this fact coupled with the high temperature coefficient of the process proves that the rate is controlled by the reaction within the fibre<sup>8-10</sup>. The uniformity of treatment obtained with chlorosulphamic acid, which gives the great permanence of the non-shrink finish (see

Part I), is also a result of a reaction controlled by fibre diffusion<sup>11</sup>. On the other hand, reaction at pH 2 with weak chlorine solutions is invariably controlled by diffusion through the solution.

### 3. THE SUPERCONTRACTION AND SOLUBILITY OF TREATED WOOL

A study of the supercontraction and solubility in dilute alkali of wool after oxidation by chloroamines showed that the wool possessed properties different from wool treated with an equivalent amount of acid chlorine.

Reaction of wool with acid solutions of chlorine does not cause supercontraction<sup>12</sup>. During treatments of wool fabric with *N*-monochlorourea or chlorosulphamic acid (> 20% available chlorine) it was observed that the patterns contracted considerably in area. This occurred in either the absence or the presence of chloride ions.

The supercontraction of bundles of wool fibres after reaction with chlorosulphamic acid in the presence of chloride ions is shown in Table VI.

TABLE VI  
Supercontraction after Treatment with Chlorosulphamic Acid

Oxidizing Agent	Available Chlorine (%) on wt. of wool)	Superecontraction (%)
Chlorine	20	0
	50	0
Chlorosulphamic acid	20	28
	50	40

It was established that solutions containing sulphuric acid, sodium chloride, and sulphamic acid in the same concentrations as were used to obtain the results given in Table VI did not cause supercontraction. The supercontraction must therefore be due to the oxidizing agent and not to the high concentrations of acid and salt in the solutions.

Wool was oxidized and then shaken with successive amounts of 0.2 N. ammonia solution in the cold until no more was dissolved. After oxidation in 10% sodium chloride solution with chlorosulphamic acid containing 50% available chlorine on the weight of wool, 95% of the wool dissolved in the alkali. However, after reaction with an equivalent quantity of chlorine in a solution at pH 1.5 only 70% dissolved. This clearly shows that the chemical nature of the reaction of chlorine with wool is different from that of chloroamines.

### 4. REACTIONS WITH MODEL SUBSTANCES

In view of the specific nature of the chloride ion effect it seemed unlikely that its function was purely physical, especially since it was seen from Table III that it did not influence the reaction of free chlorine in solution with wool. The only other possibility was that chloride ions altered the chemical reactivity of the chloroamines. To test this possibility the rate of oxidation of a number of model substances was examined. The comparative figures for reaction with chlorine are included, since they further emphasize that the reaction of chloroamines cannot proceed via a slow release of chlorine.

If this were so, the rate of reaction of chloroamines with all the model substances would be equally retarded in comparison with solutions of free chlorine. It is seen that some substances react equally rapidly with chlorine and chloroamines, whereas others react more slowly with the latter.

In Table VII are given the reaction rates, expressed as the half-lives of the reactions between *N*-monochlorourea or chlorosulphamic acid and a number of substances of structure related to that of wool. The reactions with chlorine in solution of pH 1.5 were performed at 18°C.

TABLE VII  
Reactions with Model Substances  
(Half-life in min.)

Substance	<i>N</i> -Mono-chlorourea		Chloro-sulphamic Acid		Chlorine at pH 1.5
	No NaCl	10% NaCl	No NaCl	10% NaCl	
Cysteine	1	<0.5	<0.5	<0.5	<0.5
Cystine	2	<0.5	<0.5	<0.5	1
<i>p</i> <sup>+</sup> -Diamino-diphenyl disulphide	3	<0.5	<0.5	<0.5	<0.5
Phenol	20	1	84	<0.5	<0.5
Tyrosine	70	3	∞	3	2
Silk	>100	10	80	8	4
Glycine	∞	70	∞	∞	∞
<i>α</i> -Alanine	∞	40	90	60	8
Polyglycine	∞	∞	∞	∞	∞
Polyalanine	∞	∞	∞	∞	40
Nylon	∞	∞	∞	∞	6

From this table it was concluded that the addition of salt did not influence the reaction with disulphide and peptide bonds but had a great effect on the reaction with phenolic groups such as those of tyrosine. It therefore became of immediate interest to examine the relative reaction of chloroamines with the cystine and tyrosine in wool in the presence and absence of salt.

### 5. THE OXIDATION BY CHLOROSULPHAMIC ACID OF THE CYSTEINE AND TYROSINE IN WOOL

The loss of cystine from wool after treatment with chlorosulphamic acid is given in Table VIII. The loss in weight due to the treatment was determined by drying a pattern which had been washed free from salt and neutralized by rinsing for 15 min. in dilute ammonia solution (1 ml. of sp. gr. 0.880 per 100 ml. of water).

TABLE VIII  
Loss of Cystine from Wool after Treatment with Chlorosulphamic Acid

Wt. of Available Chlorine (%)	Loss in Wt. on Treatment (%)		Cystine oxidized* (%)		
	No NaCl	10% NaCl	No NaCl	10% NaCl	10% NaCl
3.0	0	0	22.8	16.3	
5.0	1.6	2.7	24.6	22.4	
10.0	6.9	7.7	53.6	45.4	
20.0	8.8	9.8	77.5	83.1	
50.0	35.8	39.7	97.0	96.3	

\* The cystine content of the untreated wool was 10.4%, and loss of cystine was calculated from this value.

The tyrosine content of wool after reaction with chlorosulphamic acid is given in Table IX.

TABLE IX  
Loss of Tyrosine from Wool after Treatment with  
Chlorosulphamic Acid

Wt. of Available Chlorine (%)	Loss of Tyrosine (%) <sup>a</sup> No NaCl	Loss of Tyrosine (%) <sup>a</sup> 10% NaCl
3	5.50	10.3
5	6.56	18.0
10	14.54	35.1
20	46.10	68.4

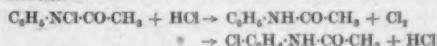
<sup>a</sup> The tyrosine content of untreated wool was 5.5%.

In agreement with the reaction of the amino acids cystine and tyrosine themselves, it is now seen that the oxidation of the disulphide bond is independent of the chloride ion concentration whereas that of phenolic groups is not.

#### 6. TREATMENT OF MODIFIED WOOL

To see whether attack on the disulphide bond was necessary to obtain non-felting, experiments were carried out by the technique of Neish and Speakman<sup>13</sup>. Wool whose disulphide bonds had been replaced by the more stable  $-\text{CH}_2-\text{S}-\text{CH}_2-$  linkages of lanthionine, which is not attacked by oxidizing agents, could not be rendered non-felting by chloroamines (Table X), and rupture of the sulphur

non-felting processes resulted from a slow hydrolysis, liberating small quantities of chlorine, and that their reaction proceeded via the intermediary of free chlorine in solution. Likhoshcerstov<sup>15</sup> regarded the interaction of *NN'*-dichlorourea and phenol as involving first the hydrolysis of the chloroamine to hypochlorous acid followed by the conversion of the latter into chlorine by means of hydrochloric acid, which then attacks the phenol. Orton and Jones<sup>16</sup> showed that the conversion of *N*-chloroacetanilide into nuclear-substituted anilides was a two-stage process involving free chlorine—



Bell<sup>17</sup>, however, came to the conclusion that in the rearrangement of *N*-bromoacetanilide in non-dissociating solvents free halogen plays no part, since the very small concentration of bromine in the reaction mixture could not react fast enough to account for the observed rate of formation of *p*-bromoacetanilide. Hurst and Soper<sup>18</sup>, studying a reaction very similar to the one reported in this paper, namely the chlorination of phenols by *N*-chloroacetanilide in aqueous solution, also concluded from the comparatively low specific rate of hydrolysis of *N*-chloroacetanilide to hypochlorous acid that the phenols reacted to a great extent by some other mechanism than that involving the intermediate production of hypochlorous acid. Similar considerations apply to the reactions studied here, but since the overall reaction with the wool is an oxidation and not a chlorination and the wool contains very little combined chlorine, there is still less need to postulate free chlorine as an intermediary in the reaction.

The following facts all indicate that the reaction between wool and chloroamines does not proceed via free chlorine in solution—(1) The concentration of free chlorine (see Table II) in equilibrium is much too low to account for the observed overall reaction rate. (2) The rate of the reaction is controlled by diffusion through the fibre with an apparent activation energy of 12 kg. cal./mole, whereas under the experimental conditions used the reaction between chlorine and wool is controlled by diffusion through a liquid film (i.e. the rate depends on stirring) with an apparent activation energy of 5 kg. cal./mole<sup>8-10</sup>. (3) The chemical reactions of chlorourea and chlorosulphamic acid are qualitatively different from those of chlorine in solution, as can be seen from their reactions with phenolic and disulphide-containing compounds (see Table VII). (4) Extensive reaction of chloroamines with wool produces supercontraction of the fibres and renders the wool almost completely soluble in dilute ammonia. After a similar reaction with chlorine solutions the fibres do not supercontract and only 70% of the wool becomes soluble in ammonia. (5) Chloride ions have a great influence on the products of the reaction, an effect not observed with aqueous chlorine solutions.

The conclusion cannot be resisted, therefore, that chloroamines themselves react with wool and that their reactions differ from those of chlorine in aqueous solution.

TABLE X  
Treatment of Modified Wool

Pretreatment	Reagent	Area Shrinkage of Pattern (%) Treated	Area Shrinkage of Pattern (%) Untreated (Control)
None	... <i>N</i> -Monochlorourea	5.3	30.4
	Chlorosulphamic acid	1.6	22.0
Acetylation	... None	26.5	21.2
	<i>N</i> -Monochlorourea	3.6	21.2
Methylation	<i>N</i> -Monochlorourea	3.5	20.7
	Chlorosulphamic acid	2.6	23.0
Lanthionine	None	18.4	20.7
formed with	<i>N</i> -Monochlorourea	14.6	20.7
NaOH	Chlorosulphamic acid	10.1	23.0
Lanthionine	None	24.9	27.4
formed by	Chlorosulphamic acid	15.6	27.4
boiling in soln. of pH 8			

cross-links also is a prime requisite for the production of non-felting with chloroamines. The acidic and basic side-groups in wool appear to take no part in felting reactions, since neither esterification of the carboxyl groups nor acetylation of the amino groups affects the reaction. The shrinkages of patterns of wool fabric which had been chemically treated prior to reaction with *N*-monochlorourea or chlorosulphamic acid, equivalent to 4.0% available chlorine, in the presence of 10% sodium chloride in solution, are given in Table X. Fabric treated with chlorosulphamic acid was hand-milled, that treated with *N*-monochlorourea was tested in a Hotpoint washing machine.

#### Discussion

The rôle of free chlorine in the reaction of chloroamines has been studied extensively. Phillips<sup>14</sup> concluded that the slow oxidation of wool by chloroamines which made them useful for

## THE NATURE OF THE REACTION

It is considered that the reactivity of a chloroamine depends on the presence of the dipole  $R-\overset{-}{N}H\cdot Cl$ . This separation of charge depends on the power of the group R to attract electrons. Hence the reactivity of the chloroamine can be seen to depend on the electronegativity of R. It was seen in Part I<sup>1</sup> that the order of effectiveness as anti-shrink agents of the different chloroamines was—

Chlorosulphamic acid > Chlorourea > Alkylchloroamines  
This is the same as the order of electronegativity of the different groups adjacent to the active  $>NCl$  group.

The great influence of chloride ions on the production of non-felting wool by chloroamines cannot result from physical factors such as decreased swelling of the fibre, since other neutral salts in equal concentrations do not influence the reaction. Moreover, if the effect were due to swelling, the rate, which is controlled by fibre diffusion, would not be independent of salt concentration. The chloride ion must influence the chemical reactivity of the chloroamines in such a way that the chemical reaction leading to non-felting is favoured. It is not possible at the moment to suggest a mechanism by which this can be brought about.

To explain these observations it is suggested that chloroamines can oxidize wool by two distinct mechanisms, neither involving the intermediary of free chlorine but only one being able to render wool non-felting. The one reaction is favoured by the presence of chloride ions and increasing negativity of the group next to the active  $>NCl$  group, and is recognized chemically by its ability to render wool non-felting, and to oxidize phenols in aqueous solution and the phenolic group of tyrosine in wool. The disulphide bond is oxidized by chloroamines whatever the mode of reaction (see Table VIII). The fact that the elastic properties of the fibre were decreased to the same extent (see Part I<sup>1</sup>) whether the reaction was carried out in the presence of chloride ions (i.e. the wool was made unshrinkable) or in their absence; when the felting power of the wool was unaffected, suggests that in both reactions the disulphide bond was severed. It seems, therefore, that attack on the disulphide bond of wool is not sufficient to bring about unshrinkability, and that some other groups such as tyrosine must be attacked also. The fact that alkali-treated wool, in which 50% of the disulphide

bonds have been converted to chemically resistant lanthionine (see Table X), cannot be rendered unshrinkable indicates that severing the disulphide bond is necessary for non-felting to occur. The present work shows, however, that this is not the only criterion, and that another point in the peptide chain, which may be tyrosine, must also be attacked. The theory of Speakman *et al.*<sup>19,20</sup> that attack on the disulphide bond of wool invariably leads to its becoming non-felting must, therefore, be modified. On this basis the function of chloride ions in the process for rendering wool non-felting with chloroamines can be seen to consist in increasing the reactivity of the oxidizing agent so as to enable it to react with phenolic groups. Although this change in chemical reactivity on adding chloride ions appears to be established both in homogeneous solution and with wool, no reason for it can be advanced.

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## The Rôle of the Disulphide Bond in Reactions which render Wool Non-felting

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It is shown from experiments with peracetic acid that breakdown of the disulphide bond alone does not remove the D.F.E. of wool fibres and thus cannot bring about non-felting by surface attack alone. Extensive disulphide bond breakdown without attack on other groups renders wool non-felting by modification of the elastic properties of the fibres as a whole. It is concluded that, to remove the D.F.E. of a fibre and to render wool unshrinkable by attack on the surface only, two reactions are necessary—firstly, as recognized by Speakman<sup>4,5</sup>, splitting of the disulphide bond without formation of a new cross-link; and secondly, breakdown of the wool molecule at another point, which in the case of the well known anti-shrink processes probably occurs at the tyrosine residue in the polypeptide chain.

### Introduction

Wool can be rendered non-felting by reaction with a large number of chemical reagents, all of which either modify the surface of the fibres or react extensively throughout the fibre to bring about a change in elastic properties<sup>1</sup>. Attack on the fibre surface can be of two kinds—it may result in loosening the whole of the scales, so that they become detached on rubbing<sup>2</sup>, or the reaction may be confined to the scale tips, which become gelatinized and glued to the underlying fibre<sup>3</sup>, and thus lose their power of bringing about the differential frictional effect (D.F.E.) necessary for felting. All commercial non-felting processes function by surface degradation, and Speakman<sup>4,5</sup> considers that all reagents capable of reacting with the disulphide bond, and in particular those which do not produce new cross-links, are effective for this purpose. Since fission of the disulphide bond brings about severe changes in the elastic properties of the fibres<sup>4</sup>, all reagents attacking this bond must bring about a measure of unshrinkability after extensive reaction, although this cannot necessarily be attributed to surface modification.

None of the oxidizing agents which render wool non-felting by surface modification confine their reaction to the disulphide bond alone, but attack other points in the molecule. Thus, detailed studies of the reactions between wool and chlorine at all pH values<sup>7,8</sup>, chloroamines<sup>9</sup>, and potassium permanganate<sup>8,10</sup> have shown that these reagents attack groups other than the disulphide bond in the wool. It is also known that chlorine dioxide, although more specific than the foregoing reagents, oxidizes tyrosine<sup>11,12</sup> as well as cystine. No detailed investigation of the reaction of proteins with sulphuryl chloride and nitrosoyl chloride has been made, but these compounds are so reactive that it is extremely unlikely that they would confine themselves only to disulphide bond attack in the wool.

Although peroxides are powerful oxidizing agents and have been claimed to render wool unshrinkable<sup>13</sup>, they do so only under drastic conditions, when the elastic properties of the fibres are severely modified and considerable damage, as shown by an alkali solubility test<sup>13</sup>, has occurred. With hydrogen peroxide, for example, Whewell *et al.*<sup>14</sup> clearly showed that after treatment for 3 hr., when the wool had acquired considerable resistance to felting, the D.F.E. was reduced by only 20% but the fibres had been gravely weakened. The D.F.E. disappeared only after an 8-hr. treatment, during which the fibre lost 50% of its strength. These

figures show that the shrinkproofing effect of hydrogen peroxide is due largely to the modification of the elastic properties, in spite of the fact that this reagent oxidizes disulphide bonds<sup>15,16</sup>, and on the basis of Speakman's theory<sup>4,5</sup> should lead to unshrinkability by the modification of the surface properties. The only conditions under which peroxides can be used for imparting a non-shrink finish without severe damage are in the presence of salts of the transition and heavy metals<sup>17</sup>, when the reactivity of the peroxide is so greatly enhanced that the process must be treated as a separate reaction.

Reducing treatments which rupture disulphide bonds irreversibly, e.g. addition of sodium bisulphite and of thiols followed by alkylation, are very poor non-felting processes. Bisulphite is completely ineffective, while thiols produce a measure of unshrinkability only after the wool has been severely degraded, as shown by the great increase in alkali solubility and loss of strength. Undoubtedly it is the change in elasticity and not surface modification which produces the resistance to milling shrinkage.

There is abundant evidence from work with wool, the disulphide bonds of which have been replaced by more stable cross links, that attack on the cystine residue is necessary for modification of the surface of the fibre to give unshrinkability<sup>5</sup>. However, in view of the fact that all chemical non-shrink agents attack other groups besides the disulphide bond, it is not evident that the breaking of the latter is the sole criterion for this reaction as suggested by Speakman. Work with chloroamines<sup>9</sup> has confirmed that it is possible to oxidize the disulphide bond without obtaining unshrinkability. It is the object of this paper to show that the complex molecular structure of wool must be broken at another point besides cystine if unshrinkability is to be brought about by surface modification.

Toennies and Homiller studied the oxidation of amino acids by performic acid<sup>18</sup> and found only methionine, cystine, and tryptophan to be attacked. This reagent, however, is unstable even at 0°C., and aqueous solutions quickly lose their oxidizing power owing to the reaction—



Peracetic acid is known to be more stable than performic acid<sup>19</sup>, and since aqueous solutions hydrolyse only very slowly it is a useful reagent for wool. Peracetic acid has been shown to exhibit the same specificity as performic acid<sup>7,10</sup>, and since

the methionine and tryptophan contents of wool are very low, it can be used for studying the rôle of the disulphide bond in the behaviour of wool.

### Experimental

#### 1. MATERIALS

The hydrogen peroxide was obtained from Laporte Chemicals Ltd. It contained 90% by weight of  $H_2O_2$ , and its great stability was due to its high purity.

Peracetic acid was prepared by interaction of 1 mole of 90% hydrogen peroxide with 1.5 moles of acetic acid in the presence of sulphuric acid, as described by Greenspan<sup>21</sup>. Within 4 hr. the system reached equilibrium at a peracetic acid content of 45%.

The wool used was 18/2 56/58s yarn knitted into fabric using 12 needles and 18 courses per inch. The scoured fabric was cut into 5-in. squares which had a dry weight of approx. 5 g.

#### 2. APPLICATION OF PERACETIC ACID TO WOOL

The preparation described above was diluted with water or carbon tetrachloride to the required concentration. After dilution with the latter, the small quantity of hydrogen peroxide and water contained in the equilibrium mixture was separated off. After immersion in the peracetic acid solution at 20°C. and a liquor ratio of 20:1 for the requisite time, the patterns were squeezed to remove excess solution and agitated in cold water containing ammonia (1 ml. of ammonia (sp. gr. 0.880) per 100 ml. of water) to remove free acid. They were then washed in cold water until neutral.

#### 3. CYSTINE ANALYSES

The cystine content of the wool was determined as described previously<sup>7</sup> using the method of Shinohara<sup>22</sup>.

#### 4. SHRINKAGE TESTING

All shrinkage testing was done by vigorous hand-milling in 1% soap solution at 30°C., the area of the pattern being measured before and after milling. An untreated control pattern was always milled together with the treated pattern. Before determining the area prior to milling, all patterns were allowed to recover from mechanical stresses, i.e. to relax, by immersion in soap solution for 1 hr.

#### 5. MEASUREMENT OF SCALINESS OF FIBRES

The violin bow method<sup>23</sup> was used and the angles of friction in the tip direction  $\theta_2$  and the root direction  $\theta_1$  were measured, both in a pH 7 buffer and in soap solution. The scaliness is expressed as—

$$\frac{\tan \theta_2 - \tan \theta_1}{\tan \theta_1} \times 100$$

#### 6. ELASTIC PROPERTIES OF THE FIBRES

The elastic properties of the fibres were investigated using a simple apparatus devised by Speakman<sup>24</sup>. From load-extension curves obtained from untreated and treated fibres the reduction in work required to stretch the fibres 30% after treatment was calculated.

### Results

#### 1. TREATMENT WITH AQUEOUS PERACETIC ACID

Results of hand-milling patterns of fabric after treatment with aqueous peracetic acid solutions of different concentrations for 30 min. are shown in Table I. A rough indication of the damage was given by the ease with which the pattern could be torn.

TABLE I  
Treatment with Aqueous Peracetic Acid Solutions of Different Concentrations

Concentration of Peracetic Acid (%)	Area Shrinkage (%)	Damage
0	26.1	—
1.0	15.8	Very slight
2.0	12.2	Very slight
3.0	4.3	Severe degradation
4.0	—10.6	Severe degradation
5.0	—	Disintegrated in milling

The cystine content of wool after treatment with 3.0% aqueous peracetic acid for different times is shown in Table II.

TABLE II  
Cystine Content of Wool after Treatment with 3.0% Aqueous Peracetic Acid

Time of Treatment (min.)	Cystine Oxidized* (%)
0	0
1	29.7
5	38.6
60	81.8

\* The cystine content of the untreated wool was 10.4%.

These preliminary experiments established that peracetic acid very rapidly reacted with the cystine residue of wool, but that the wool was rendered non-felting only after extensive reaction, which weakened the fibres as a whole.

A more comprehensive series of experiments using a 1.6% aqueous solution of peracetic acid for different times was carried out, and the loss of cystine, scaliness, elasticity, and shrinkage of the wool are correlated in Table III.

TABLE III  
Treatment of Wool with 1.6% Aqueous Peracetic Acid

Time of Treatment (min.)	Cystine oxidized (%)	Reduction in Work		D.F.E. (%)	Area of Pattern (%)
		30% in 0.1N-HCl pH 7	30% in pH 7		
0	0	39	2	80	30 26.7
5	19.8	48	28	55	28 23.4
				51	37
30	46.3	70	67	39	31 8.8
				41	39
60	71.1	82	84	30	22 4.0
				30	28
120	75.0	93	97	22	9.7
				25	Disintegrated

It is evident from these results that peracetic acid is not able to remove the D.F.E. of wool fibres and that felting resistance is almost entirely due to the severe reduction in their elasticity.

## 2. APPLICATION OF PERACETIC ACID FROM NON-AQUEOUS SOLVENTS

Wool becomes soluble in dilute alkalis such as soap solution or 0.3 N. ammonium hydroxide when all the disulphide bonds have been oxidized<sup>23,24</sup>. It was thought possible that by applying peracetic acid from a non-swelling solvent the oxidation could be confined mainly to the surface, and that it should be possible in this way to solubilize the cuticle but not the cortex. It is seen from Tables IV and V that this is in fact possible and that the wool becomes unshrinkable. It was observed by careful microscopic examination that the whole of the scale structure of the fibre disappeared in soap solution by dissolution: no loose scales could be seen to come off the fibres, as was shown by photomicrographs in the case of chlorination<sup>9</sup>. When the scales had been dissolved, the fibre, as was to be expected, no longer possessed a D.F.E.

TABLE IV

Treatment of Wool at 14% Regain for 10 min. with Peracetic Acid in Carbon Tetrachloride of Varying Concentrations

Peracetic Acid Concentration (% wt./vol.)	Area Shrinkage of Pattern (%)
0	22.6
0.5	2.3
1.0	4.6
1.5	0.4 (severely degraded)

TABLE V

Treatment with a Solution of 0.50% (wt./vol.) Peracetic Acid in Carbon Tetrachloride for Different Times

Time of Treatment (min.)	Area Shrinkage of Pattern (%)
0	25.1
3	13.2
6	7.3
9	3.7

The optimum conditions for rendering wool of 14% regain non-felting using carbon tetrachloride was a treatment for 9 min. in 0.5% peracetic acid solution at 20°C. A similar treatment was necessary when white spirit was used as the solvent. Although the whole of the cuticle is removed, the reaction has been largely confined to the surface, as the alkali solubility of the wool<sup>13</sup> increased only from 10 to 27%. When a similar resistance to felting is obtained with aqueous solutions of chlorine the alkali solubility of the wool is approx. 20%. From analysis of the bath before and after treatment it was found that the wool had consumed 3.3% of peracetic acid calculated on its weight.

## 3. TREATMENT WITH HYDROGEN PEROXIDE IN NON-AQUEOUS SOLVENTS

It seemed important to determine whether hydrogen peroxide when applied from non-swelling solvents is capable of rendering wool unshrinkable by surface modification. The extensive reaction resulting in change in elastic properties which occurs in aqueous solutions is obviously less likely under these conditions.

Patterns of wool fabric were treated for 3 hr. at 20°C. with hydrogen peroxide dissolved in ether or isopropyl, *tert*-butyl, or *n*-amyl alcohol. In no case did reduction in felting occur, although solutions containing up to 9% H<sub>2</sub>O<sub>2</sub> were used.

Further treatments were then performed at 80°C. The patterns were treated for 3 hr. with hydrogen peroxide dissolved in *n*-amyl alcohol using a liquor ratio of 20 : 1. The variation of the hand-milling shrinkages with concentration of solution is shown in Table VI.

TABLE VI  
Treatment with Hydrogen Peroxide in *n*-Amyl Alcohol at 80°C.

Concentration (% H <sub>2</sub> O <sub>2</sub> wt./vol.)	Area Shrinkage of Pattern (%)	Treated	Untreated
0.66	18.0	28.0	
1.10	20.4	26.6	
1.39	6.5	23.9	
3.40		Disintegrated	

All the patterns were severely degraded, and it is thus seen that the results are very similar to those of Whewell<sup>14</sup>, unshrinkability being obtained only after severe damage. It appears that hydrogen peroxide cannot bring about resistance to felting by surface modification under any conditions.

## 4. TREATMENT WITH AQUEOUS PERCHROMIC AND PERVERANADIC ACIDS

Perchromic and pervanadic acids were examined to see whether, in their reaction with wool, they behaved similarly to hydrogen peroxide or to the organic peracids.

Wool fabric (10 g.) was treated with 800 ml. of water containing 2.5 g. of potassium chromate or ammonium vanadate, 2 ml. of concentrated sulphuric acid, and 2.5 ml. of 30% hydrogen peroxide solution for 3 hr. at 20°C. Under these conditions the solution contained the peracids HCrO<sub>5</sub> and HVO<sub>4</sub>. After treatment the wool was badly discoloured. Results of hand-milling the patterns are shown in Table VII.

TABLE VII  
Treatment of Wool with Perchromic and Pervanadic Acids

Reagent	Area Shrinkage of Pattern (%)
None	19.8
	20.5
Perchromic acid (20°C.)	18.7
	20.9
Pervanadic acid (20°C.)	20.8
	24.8
Pervanadic acid (60°C.)	21.6
	19.4

These inorganic peracids are obviously not nearly as reactive as the organic peracids towards wool and appear to behave like solutions of hydrogen peroxide.

## Discussion

Experiments in aqueous solution with the highly specific oxidizing agent peracetic acid have shown that it is not capable of removing the D.F.E. of wool fibres. Wool can be made unshrinkable with

aqueous solutions of peracetic acid, but only after severe degradation which results in loss of elasticity. Admittedly, peracetic acid from organic solvents, by completely solubilizing the cuticle of the wool, is able in this way to render wool non-felting, since a fibre without scales has no D.F.E. Under similar conditions hydrogen peroxide, which also oxidizes the disulphide bond, fails to bring about unshrinkability since it cannot bring about complete dissolution. Dilute acid solutions of chlorine<sup>27</sup> and 1.6% peracetic acid solution<sup>7</sup> react with wool at roughly the same rate and oxidize 14% of the cystine in 2-3 min. at 20°C. Yet this amount of reaction with chlorine (3% on the weight of wool) completely removes the D.F.E. of the fibres, whereas the peracetic acid hardly reduces it. Short, therefore, of complete dissolution of the cuticle, there is no evidence that oxidation of the disulphide bond alone can bring about the degree of surface modification necessary for rendering wool non-felting.

The conclusion cannot be resisted, therefore, that the effectiveness of chlorine, acid solutions of permanganate, chlorine dioxide<sup>28</sup> (and also sodium chloride<sup>29</sup>), chloroamides, and chlorosulphonamides as non-felting agents must be the result of the breaking of another part of the wool molecule in addition to that of the disulphide bond.

It seems probable that for the reagents enumerated above the other point of attack is at the tyrosine residue for the following reasons<sup>4</sup>—

(1) Although chlorine is capable of oxidizing all the amino acids of wool<sup>30</sup>, it reacts preferentially with tyrosine and cystine, and in wool oxidizes the other amino acid residues only very slowly<sup>4</sup>.

(2) Permanganate at pH 2 reacts rapidly with cystine and tyrosine, but not detectably with other individual amino acids at room temperature<sup>10</sup>. In wool, acid permanganate does attack amino acid residues other than cystine and tyrosine, but only very slowly.

(3) Schmidt and Braunsdorf<sup>11</sup> showed that chlorine dioxide oxidized only tyrosine and cystine, and it appears to be specific for these amino acids even when they are combined in proteins. Speakman and Das<sup>28</sup> confirmed that this reagent confined its attack to these two amino acids in wool.

(4) Chloroamides and chlorosulphonamides were shown by the present authors<sup>9</sup> to render wool unshrinkable much more readily in the presence of a high concentration of chloride ions, the presence of which was shown from analytical data not to influence the reaction with the disulphide bond but to promote the reaction with phenolic groups such as the tyrosine residue in wool. The necessity for oxidizing a group other than the disulphide bond was demonstrated unambiguously with chlorosulphamic acid, since, using the equivalent of 3% of chlorine on the weight of wool, the rate of reaction and the quantity of cystine oxidized were approximately the same in the

presence and absence of salt and yet only in the former case was the wool rendered non-felting. The only difference was that in the presence of salt the tyrosine content of the wool was reduced by 10.3% as opposed to only 5.5% in the absence of salt.

Further support for the suggestion that two sites have to be attacked to make wool non-felting comes from a recent patent<sup>31</sup>, in which it is stated that wool can be made resistant to shrinking by the combined action of a reducing agent such as a bisulphite and an alkaline solution of permanganate. Neither reagent by itself can bring about non-felting under the conditions used. Phillips<sup>31</sup> showed that bisulphite ruptures the disulphide bond, whereas alkaline permanganate does not react with this group except after very extensive reaction but readily oxidizes the tyrosine in wool<sup>20</sup>. In this process also it seems reasonable to identify the production of non-felting with attack on the cystine and tyrosine in wool.

Although oxidation of the tyrosine residue is thus seen to play an important part in the reaction of the common anti-shrink reagents, attack on this group alone does not lead to the loss of the D.F.E. of wool fibres. Thus, solutions of potassium permanganate at pH 9<sup>20</sup> and nitric acid<sup>32</sup>, which readily oxidize the tyrosine in wool but do not react with the cystine, do not render wool unshrinkable until major chain degradation leading to loss of elasticity has occurred.

In view of the finding that hypochlorite was capable of reacting with both cystine<sup>7</sup> and tyrosine<sup>33</sup> in wool, it was difficult to understand why solutions of chlorine of pH above 9<sup>3</sup> were so much less effective in rendering wool unshrinkable than chlorine solutions at lower pH values. A detailed study by Alexander, Fox, and Hudson<sup>8</sup> of the oxidation products of cystine in wool has provided a probable explanation. Chlorine in solutions of pH less than 8 ruptures the disulphide bond with the formation of two sulpho groups. At pH 10, however, the hypochlorite reacts exclusively to form lanthionine (i.e. the  $-\text{CH}_2-\text{S}-\text{S}-\text{CH}_2-$  cross-link) is replaced by a  $-\text{CH}_2-\text{S}-\text{CH}_2-$  cross-link). The reaction is definitely due to the hypochlorite and not the alkalinity of the solution, since no disulphide bond reaction occurs at pH 10 in the absence of hypochlorite under the conditions used (1 hr. at room temperature).

From the foregoing it can be concluded that to remove the D.F.E. of a wool fibre (i.e. to render it non-felting by surface modification) not only is it necessary to break the disulphide bond in such a way that a new cross-link is not formed, but in addition it is also necessary to break the wool protein at another point in the structure. With the common anti-shrink agents used the other group attacked appears to be the tyrosine residue, probably because it is one of the most reactive side-groups present in reasonable quantity. The most useful working hypothesis of Speakman<sup>4</sup> that “any oxidizing agent capable of causing disulphide bond breakdown is *ipso facto* a potential agent for producing unshrinkability” therefore requires modification.

\* Tryptophan and methionine have not been included in this survey since they are present only in very small amounts in wool. They are oxidized rapidly by all the oxidizing agents discussed, but their reaction is not thought to be significant.

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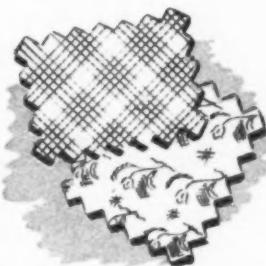
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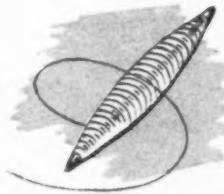
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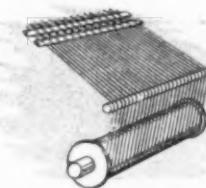
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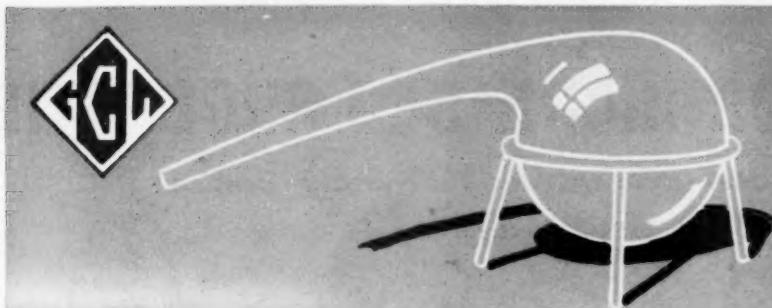
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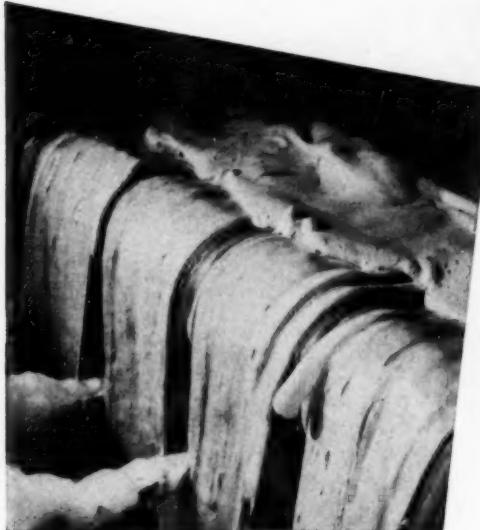
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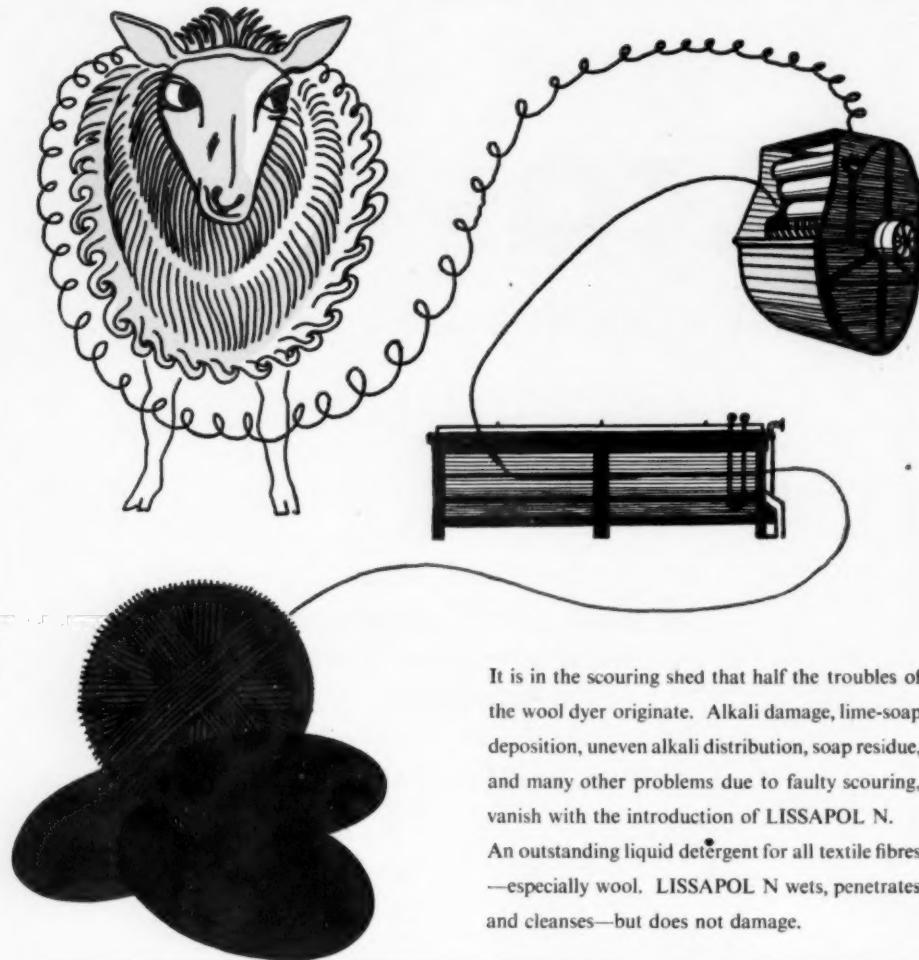
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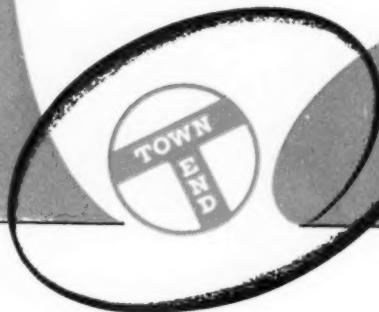
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The only apparent exception to this rule is fluorine. Hudson and Alexander<sup>23</sup> found that in the initial stage of the reaction of wool with fluorine the only reaction product was sulphur hexafluoride, although the wool was rendered unshrinkable. As attack on groups other than disulphide would give carbon fluorides or hydrogen fluoride, it was concluded that the initial action was confined to this group only. The whole process was, however, studied in a gaseous flow system, and only reaction products leaving the wool were looked for. Any hydrogen fluoride produced, for example, by oxidizing tyrosine could have readily been retained by the wool, either by hydrogen bonding in the same way as hydrogen peroxide is sorbed<sup>24</sup> or by combination as an acid on the salt-links. The experiments with fluorine cannot, therefore, be considered as an argument against the general picture proposed, which is based on detailed analytical examination.

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## CORRESPONDENCE

*The Editor does not hold himself responsible for opinions expressed by correspondents*

**The Editor**  
Sir,

I refer to the paper by Alexander, Carter, and Earland in your October issue, *Reaction of Wool with Chloroamines—I*, and particularly to Table XXI on p. 543. Whilst the latent damage test is doubtless of considerable value, it appears to me that the method of presentation is misleading. Under the heading "Latent Damage (%)" high

figures such as 91 indicate only very slight damage, whereas low figures such as 41 indicate very heavy damage. Perhaps if "Latent Damage (%)" were expressed as 100—91, and 100—41, i.e. 9 and 59 respectively, a clearer picture would be presented.

Yours faithfully

R. W. MONCRIEFF

3 CHURCH SQUARE  
HARROGATE  
11th November 1950

## Notes

### Proceedings of the Council

At meetings of the Council, held at the offices of the Society, 32–34 Piccadilly, Bradford, on 11th October and 8th November 1950, the proceedings included the following items of interest—

**SUMMER SCHOOL IN LEEDS**—It was resolved that the surplus of £50, shown by the final accounts of the Summer School, should be given to the Department of Colour Chemistry and Dyeing of the University of Leeds as a token of the Society's appreciation of the willing co-operation of the Department and in view of the extra expenditure indirectly incurred by the Department in connection with the Summer School.

**WOOL DYES COMMITTEE**—It was resolved that the Committee on the Dyeing Properties of Wool Dyes be dissolved and that the members be cordially thanked for their services.

**DYERS' CO. MEDAL**—The report of the Adjudication Committee for the Research Medal of the Worshipful Company of Dyers (see p. 5) was accepted.

**PRESIDENTIAL BADGE**—Designs for a Presidential Badge of Office, which the Worshipful Company of Dyers had very generously agreed to present to the Society, had been considered by a committee consisting of Messrs. G. B. Angus, J. Barritt, G. E. Holden, G. G. Hopkinson, and J. M. Preston. A design by Mr. J. Chirnside, based on a regular

hexagon within a vertical gold ellipse, submitted by the Committee, was accepted by Council and subsequently by the Court of the Company. The very grateful thanks of Council were expressed to the Company, the Committee, and Mr. Chirnside.

**FELTMAKERS' CO. MEDAL**—The report of the Adjudication Committee for the Research Medal of the Worshipful Company of Feltmakers (see p. 6) was accepted.

**MEMBERSHIP**—Twenty-eight applications for ordinary membership and ten for junior membership were approved.

**BELGIAN TEXTILE CONGRESS**—Mr. J. Barritt reported that he had conveyed the good wishes of the Society to the Belgian Textile Society when he had attended their Annual Conference in Brussels on 16-18th October 1950.

**LEEDS JUNIOR BRANCH**—It was reported that at the Annual General Meeting of the Branch the following officers had been elected—

*Chairman*—Mr. C. L. Bird.  
*Vice-Chairman*—Professor W. Bradley  
*Honorary Secretary*—Mr. C. R. Thitchener  
*Committee*—Messrs. D. Barton, W. B. Ingleby, E. R. Inman, and D. A. Wade

#### Meetings of Council and Committees December 1950

Council—13th  
 Finance—13th  
 Publications—19th  
*Colour Index* Editorial Panel—4th  
 Fastness Tests Co-ordinating—8th  
 Terms and Definitions—21st  
 1951 Conference—15th

#### Worshipful Company of Feltmakers' Research Medal

The Adjudication Committee report that they again feel unable to recommend the award of the medal, for the period July 1949 to June 1950.

Both the Committee and the Council are very disappointed at the scarcity of papers on the subject of feltmaking appearing in the *Journal*, and they feel that the existence of the medal should be more widely publicized. The Feltmakers' Company offer annually a Gold Medal for papers embodying the results of scientific research or technical investigation connected with the art of feltmaking and published in the *Journal* of the Society.

## OBITUARY NOTICE

### Ernest Bryan Adams

After graduation at Birmingham University followed by a period of post-graduate research under Professor Frankland, E. B. Adams entered industry in 1909 when he joined Messrs. Curtiss & Harvey, explosives manufacturers. When the first world war came, he was associated with a large extension of this company's factory at Cliffe-at-Hoo, Kent, and assumed control of the large output of naval cordite.

After the war he took up an appointment in the Research Department of Messrs. Levinstein of Manchester, dyemakers, now a part of I.C.I., and envisaged the development of a dyemaking industry in this country—a vision which never left him and to the materialization of which his subsequent experience enabled him to contribute to no small extent. After a short period with Messrs. Mandelberg, where he was concerned with the application of dyes, he became associated with the English Sewing Cotton Co. Ltd. in 1925. Following a short term as Assistant, he took over the post of Chief Chemist, and acted in this capacity until his death. He was responsible for many of the developments which have taken place in the wet

processing departments of the company during the past twenty-five years.

During the war years he served on various technical committees set up to deal with the problems connected with supplies of yarn for military purposes generally. These were indeed strenuous days, but his strong civic sense compelled him to undertake heavy duties in civil defence. Loss of his elder son, a pilot in the R.A.F., was a deepseated and lasting blow to him.

He was a member of the Society for thirty years, during which he gave freely of his time in the common interest. He not only took an active part in the Manchester Section, being a member of the committee from 1931 to 1947 and a vice-chairman from 1934 to 1936, but was a valued member of both the Direct Cotton Dyes and the Vat Dyes Committees. His wide experience, sound judgment, and charm of manner were of the greatest help in solving the many problems which arose.

Intense interest in his work and a capacity for hard work were characteristic throughout his career. He died in harness as he would have wished, leaving an inspiring memory of a sterling and lovable character.

C. BARROW  
 P. W. CUNLIFFE

## New Books and Publications

### Injuries and Accident Causes in Textile Dyeing and Finishing

United States Department of Labor, Bureau of Labor Statistics. Bulletin No. 962. Pp. vi + 65. Washington, D.C.: U.S. Government Printing Office 1949. Price, 45 cents.

Compiled as a "Detailed Analysis of Hazards and 1945 Injury-Frequency Rates by Region, Size of Plant, and Occupation", the bulletin commences with a record of the injury frequency rates of the industry over a number of years, indicating that in comparison with other American industries the

dyeing and finishing industry is losing ground, which leads to the conclusion that accident prevention probably has been given less attention than in other industries.

Then follows an interesting survey of the industry and its hazards, which gives a very general idea of American methods and practices. In this survey it is recorded that "woollen cloths are generally run through a 'boil off' machine in which they are treated with caustic soda"—a rather remarkable statement which gives the impression that First Aid may on occasion not only be required by the operative. Next are given a broad review of types of injuries, and an analysis of accidents and their causes, together with block graphs and suggested methods for attacking the problems enumerated. After a description of typical dyeing and finishing accidents, the bulletin finally concludes with 30 tables analysing accident statistics supplied from voluntary reports by 446 plants covering most aspects of the industry.

It is rather remarkable that none of these tables indicates the relation of accidents either to years of service or to age. In one concern in this country in which accident statistics have been compiled over a number of years, it has been found that on average 50% of all accidents occur within the first two years of service, and that 38% of all dermatitis cases occur within the same period. As regards age groups, it is interesting to note that the peak curve is reached between the ages of 46 and 50 years, probably owing to the fact that comparatively few juniors are entering the industry at the present time. Another interesting point is that 59% of all accidents appear to occur to hands and fingers.

The bulletin rightly stresses the advantages of accident prevention committees, adding that, regardless of size, plants in which the management take a genuine interest in safety measures invariably have the best accident records. In the small plant economics bars the employment of a whole-time safety expert, but the appointment of one worker in each department to take the newcomer under his or her wing, stressing the dangers likely to be encountered, and supplying details of the first-aid schemes, etc., will be found nearly as efficient. This, coupled with an active accident prevention committee and a weekly inspection of hands by the first-aid staff, will tend to reduce accidents and dermatitis to comparatively low levels.

H. JENNISON

#### History of Dyes and Dyeing in the Bombay Presidency

By B. N. Phadké. Pp. xx + 152. Poona: Dastane Brothers' Home Service Ltd. [1947]. Price, Rs. 12-0-0.

After a short historical introduction in which the antiquity of dyeing in India is discussed, the author describes the application of dyestuffs indigenous to the Bombay Province. Although these natural dyestuffs have been largely replaced by synthetic dyes, they are still used to a very small extent, and nearly half the book is devoted to their properties and applications. The pretreatment of cotton and subsequent dyeing with natural colouring matters to produce a full range of shades is dealt with in

considerable detail; the methods of application of the dyestuffs to silk and wool is described, although little wool dyeing is actually carried out. Details are given for dyeing cotton with *kasumba* or safflower, which is almost an entirely separate branch of the dyeing industry. Other sections deal with calico printing (block printing is extensively practised), knot dyeing, silk printing (carried out only to a small extent), ivory and wood dyeing, and the use of mineral pigments for colouring textiles with especial reference to the production of mineral and vegetable khaki shades.

The remainder of the book deals mainly with the application of synthetic dyes to cotton, wool, silk, and viscose rayon, indications being given of the extent to which the various classes of dyes and any special methods, e.g. aftertreatments, are used in the Presidency.

A short historical review of the gradual replacement of natural by synthetic dyes is included in an Appendix, together with various tables and some maps showing important dyeing and calico printing centres formerly engaged in the application of indigenous dyestuffs. The varieties of textile goods now manufactured at mills in the Presidency and the types of dyes used are also tabulated.

There are many spelling and minor errors, e.g. "diaz" for *disazo*, "Chrysophene" for *Chrysophenine*, and some incorrect statements, e.g. that the *Algols* and *Hydron* dyes are indigoid derivatives. In the section dealing with wood dyeing, the distinction between dyeing and painting is not drawn, and it is difficult to understand what is intended by such a statement as "dyeing the wood with pounded or melted tin" whatever treatment is subsequently applied. Some errors occur in the list of natural dyes (Appendix, Table I)—*Alizarin* is stated to be the colouring matter present in *Munjeet* and in *Morinda Root*; whereas the former contains purpurin and munjistin, and the latter morindone. Red *Sanderswood* owes its dyeing property to *santalum* and *deoxysantalum*, not to *homopteroarpin* as stated. *isoRhamnetin* (quercetin 3'-methyl ether) is incorrectly given as a derivative of naphthaquinone, whilst catechin, present in catechu, is the 2-, and not the 4-phenylchroman derivative cited. More precise information could have been included in some cases; e.g. lawsone is present in *Henna* and is 2-hydroxy-1:4-naphthaquinone, and nyctanthin, obtained from *Toon* flowers, is identical with crocetin.

The book is chiefly interesting for the information it contains concerning the application of natural dyes.

E. J. CROSS

#### DDT and Newer Persistent Insecticides

By T. F. West and G. A. Campbell. 2nd edition revised 1950. Pp. xv + 632, with 13 plates. London: Chapman & Hall Ltd. Price, cloth, 50s. Od.

This book, the first edition of which was published in 1945 under the title *DDT: the Synthetic Insecticide*, is divided into two parts, the first of which (pp. 1-497) deals solely with DDT, while the second (pp. 501-568) deals with benzene hexachloride, chlordane, and several products of which

as yet little is known. So far the products discussed in the second part of the book have found no application of direct concern to tinctorial technologists or textile chemists.

The first part of the book has a prologue which deals with the history and development of the use of DDT, followed by chapters dealing in detail with every conceivable property and use of DDT. Chapter I is of particular interest, as it is not only a valuable contribution to the literature on the chemistry of mothproofing but is also an excellent illustration both of how research in one field can lead to unexpected and revolutionary developments in entirely different fields and of how attention should be paid to the observations of even the most unscientific observers. It was noticed by German housewives of the 1870s that many cloths dyed green did not become moth-eaten, and so the saying arose among them that green cloths in general were not attacked by moths. Many years later a research chemist to whom this saying was familiar noticed a green-covered sofa that was moth-eaten, and this set him to work to find out what basis there was for the old wives' tale. His work led him to discover the mothproofing properties of inorganic fluorides, and this in turn led his colleagues at Leverkusen to devise organic mothproofing agents. By 1932 the sale of such products by the I.G. had become substantial enough to set a rival firm of chemical manufacturers to work to find products which could claim a share of the market that had been created. In efforts to discover a benzene-soluble mothproofing agent to compete with the I.G.'s Eulan BL, Müller in the Geigy laboratories discovered the insecticidal properties of DDT, a compound which, although known for 70 years, had not till then been tried in that direction. The subsequent history of DDT, known in outline to everyone, is detailed in this book. All honour is due to Müller for this discovery, but would he ever have had the opportunity of making it if some unknown housewives 60 or more years earlier had not noticed that some green cloths were immune to moth attack? When the third edition of this book appears, and a third edition will be surely called for, it could have a much worse dedication than to the memory of those unknown women.

Other chapters of direct interest to members of this Society are Chapter II on the manufacture and chemistry of DDT and its analysis on other materials, Chapter V dealing with its use in paints, Chapter VI dealing with its use in the textile and paper industries, and Chapter X dealing with its use against household pests, many of which are also pests in our warehouses and dyehouses. The other chapters are concerned with the use of DDT in the agricultural and horticultural, veterinary and pharmaceutical fields, as is only right much attention being paid to the revolution it has wrought in the campaigns against houseflies, mosquitoes, and human lice.

The book is well written with a wealth of detail and references to the literature, an appendix bringing the literature references up to 1948. There are author and subject indexes and also an index

of systematic names of insects. As the authoritative account of one of the most important discoveries ever made by research chemists and its successful practical application, the book will be of much service to all concerned with the protection of textiles and other stored products, plants, and animals from attack by insects. C. O. CLARK

#### Organic Reagents for Organic Analysis

By the Staff of the Research Laboratory, Hopkin & Williams Ltd. 2nd edition 1950. Pp. 263. Chadwell Heath, Essex: Hopkin & Williams Ltd. Price, 12s. 6d.

A review of the first edition of this book (1944) appeared in the *Journal* five years ago (J.S.D.C., 62, 218 (July 1946)). For convenience, it may be recalled here that the book is divided into three main sections—

(1) A general survey, classified under types of substances for identification, covering about seventeen groups, such as carboxylic and sulphonic acids, aldehydes, ketones, amines, amides, ethers, halides, aromatic hydrocarbons, thiols, phenols, etc. For each group there is a critical discussion of the various reagents available.

(2) A list of selected reagents, and recommended methods for the preparation of derivatives.

(3) Tables of melting points of the derivatives.

The second edition follows the first very closely and is essentially a revision and extension. It is, in fact, more than 50% larger than the first edition; the biggest increase is in the tables of melting points of the derivatives, which now occupy double the number of pages. In this connection the authors acknowledge their indebtedness to Heilbron and Bunbury's *Dictionary of Organic Compounds*.

From a survey of the book it is clear that its compilers have spared no efforts in making its various sections, including bibliography, as up-to-date as possible.

An essential feature of the book is that it is by no means a mere catalogue but is a critical survey, based largely on the work of the authors and their collaborators. Arising from this there are "selected reagents" and "other reagents", and an adequate discussion of the relative merits of the two types.

In the second edition there are some half-dozen additional "selected reagents". These are used for the identification of aldehydes, alkyl halides, esters of aliphatic dibasic acids, conjugated dienes, polycyclic hydrocarbons, and furan derivatives.

Perhaps the greatest value of the book lies in the fact that general working details are given for the preparation of derivatives from the "selected reagents". The reviewer feels that these little "tricks of the trade" in making what are, after all, "organic preps." on the small scale should be of the greatest help to an analyst who is called on to identify technical organic products.

In the first review it was stated that "It is well produced, at a very reasonable price, and can be recommended without hesitation to the organic analyst and research worker." It can now be said with equal confidence that these same workers would find the purchase of a copy of the second edition to be a good investment. N. STRAFFORD

**Synthetische Methoden der Organischen Chemie**

**Repertorium 4**

By W. Theilheimer. Pp. xi + 360. Basle and New York: S. Karger. 1950. Price, 37 Swiss francs.

The present volume 4, though still issued only in a German edition, admirably continues the collection of new organic synthetic methods, improvements of known methods, and also old proved processes which are scattered about in the original papers of the periodicals. This is now done over the period of one year with the classification adopted in previous volumes (cf. *J.S.D.C.*, **64**, 399 (Dec. 1948); **66**, 328 (June 1950)), and in effect contains abstracts from papers published in 1947 and 1948 with supplementary references from 1949. A cumulative list of the latter is provided. Reaction titles from preceding volumes have been included only when new references have been added. The

scope of this volume may be realized from the fact that 865 methods have been included in its 316 pages, all being accompanied by formulae where necessary, which greatly facilitate the interpretation of the concise abstract. Syntheses that are split into their various steps, which are recorded in different places, can be followed with the help of a supplied notation. The abstracts now collected are limited to the information needed for an appraisal of the applicability of a desired synthesis, so that it is still necessary to have recourse to the original papers or, at least, to an abstracting journal. Continuations of papers already included are not abstracted unless they contain new data. Less accessible and readable papers, such as the Russian ones, are abstracted only if the method in question is described nowhere else. An index key in English has been included, but it is stated that volume 5 will appear in English. The book can be recommended to all organic research chemists.

H. H. HODGSON

## Reports on German Industry

**I.G. Farbenindustrie A.-G., Leverkusen**  
**Intermediates for Dyes and Plastics**  
**Mothproofing Agents**

FDX 877\* (PB 82,165; Microfilm N 81).

Numerous reports (857 pp.) on work done during 1929-44, mostly on intermediates. There are 15 reports on work done during 1932-35 on mothproofing agents. C. O. C.

**I.G. Farbenindustrie A.-G., Fechenheim,**  
**Frankfurt a. M.**

**Dyes and Intermediates—Auxiliary Agents**

FDX 878\* (PB 84,316; Microfilm D 209).

Methods of manufacture together with structural formulae are given of numerous intermediates, dyes, and auxiliary agents. The dyes include—

Algol Orange RF and RF Suprafix, Violet BBN, Yellow GR

Citroflavin 8G

Crystal Ponceau 6R, special, and other brands

Hydron Printing Blue 3R, Violet N, Yellow GG

Immedial Olive FF; Red Brown CL3R; Yellow GG, 3GT, and RR

Indanthren Brilliant Orange GE [?GR], GK, and RK; Brilliant Pink 3B, R, and R Suprafix; Brown NG; Olive GB; Printing Olive GW Suprafix; Printing Pink FFB and FFR; Printing Red 3B; Printing Red Violet RRN; Printing Violet BBF; Red Brown 2R; Red Violet RRN; Rubin R; Scarlet B

Indocarbon IB Suprafix and IR Suprafix

Primulin ECD

Sirius Supra Yellow FRL, GG, RR, and RT

\* Material available for inspection at, and photocopies available from, T.I.D.U., Board of Trade, Lacon House, Theobalds Road, London W.C.1, or (under PB No.) from Office of Technical Services, Department of Commerce, Washington D.C., U.S.A.

Thioflavin S and TCN

Van Brown BT and IVD, Scarlet RM, Violet RR.

Instructions for standardizing Algol and Indanthren powder brands are also given. Among the auxiliary agents are the following—Azamin (melamine); Kaurit MKF; Katanol O, ON, and SL; Solidogen B and BSE; Uren AN and BR. C. O. C.

**I.G. Farbenindustrie A.-G., Leverkusen**

**Reports on Dye Research 1937-43**

FDX 856\* (PB 74,894; Microfilm 30 T).

Monthly, quarterly, and annual reports (747 pp.) on work on dyes are given for 1937-43 together with a few reports on work on production of diisocyanates and other organic compounds, polymers, etc. C. O. C.

**I.G. Farbenindustrie A.-G., Ludwigshafen**  
**Analysis—Preparation of Organic Compounds,**

**Resins, and Plastics**

FDX 880\* (PB 74,709; Microfilm 142 M).

Some 26 methods of inorganic and organic analysis are given, together with a number of reports on the manufacture of various organic compounds, resins, softeners, and plasticizers. C. O. C.

**[Ceramic Colours and Lithographic Transfers used in the Fine Ceramics Industry in Germany during 1939-45]**

D. A. Holdridge. BIOS Survey No. 28. H.M.S.O. Price, 3s. 6d.

Pp. 124 and 125 of this survey summarize the information scattered in the various BIOS, FIAT, etc. reports and give references not only to these reports but also to FD documents. C. O. C.

## Abstracts from British and Foreign Journals and Patents

(The Titles of Patents are abridged and modified)

### Abbreviations

**BrC** British Celanese Ltd. **CAC** Clayton Aniline Co. Ltd. **Ciba** Ciba Ltd. **CN** Compagnie Nationale de Matières Colorantes et de Produits Chimiques du Nord Réunies Etablissements Kuhlmann. **DH** Durand & Huguenin A.-G. **duP** E. I. du Pont de Nemours & Co. **Gy** A. R. Geigy A.-G. **ICI** Imperial Chemical Industries Ltd. **IG** I.G. Farbenindustrie A.-G. **LBH** L. B. Holliday & Co. Ltd. **S** Sandoz A.-G. **YDC** Yorkshire Dyeware & Chemical Co. Ltd.

The above abbreviations are used after the names of commercial dyes. The following abbreviations are used in the titles of abstracts of patent specifications—**Kuhlmann** Compagnie Nationale de Matières Colorantes et de Produits Chimiques du Nord Réunies Etablissements Kuhlmann. **du Pont** E. I. du Pont de Nemours & Co. **L.C.I. Ltd.** Imperial Chemical Industries Ltd. **Sandoz** Sandoz A.-G.

### I—PLANT; MACHINERY; BUILDINGS

#### PATENTS

**Continuous Aftertreatment of Rayon.** H. A. Schrenk and American Enka Corp. *U.S.P. 2,513,057.*

In the continuous production of rayon yarn the component filaments of the yarn are separated so as to cause the yarn to assume a ribbon form during its passage through the aftertreatment liquor. This ensures more thorough and uniform treatment of the individual filaments. *C. O. C.*

**Continuous Treatment of Filaments by Liquid.** J. A. Truitt and American Viscose Corp. *U.S.P. 2,513,381.*

**Continuous Treatment of Fibrous or Filamentary Material.** W. A. Sisson and American Viscose Corp. *U.S.P. 2,513,432.*

**Providing a Continuous Indication dependent on the Tension in Material passing through a Machine.** A. Ashbury, G. R. Wilson, and English Electric Co. Ltd. *B.P. 643,775.*

**Measuring the Length of Material passing through a Machine.** H. Burr, G. T. W. Hall, L. W. J. Harris, and Standard Telephones & Cables Ltd. *B.P. 643,838.*

**Dyeing Yarn on Cones.** W. D. MacNeill. *U.S.P. 2,513,418.*

The paper or other supporting cone on which the yarn is wound is withdrawn from the package, without disturbing the yarn convolutions, and replaced with a perforated metal cone of the same conical form and approx. size as the first cone but longer and somewhat outwardly flared for about 0.75 in. near its smaller end. The cone is then introduced into a suitable dye vessel, a preferred form of which is described. *C. O. C.*

**Hank Drying.** J. Dean. *B.P. 644,407.*

The hanks are carried through the drying chamber on swivelling hooks borne by an endless chain. The hooks have extending parts which strike fixed abutments, so that, at the loading and unloading points, the hooks extend at right-angles to the chain to facilitate loading and unloading, and while in the drying chamber they are in line with the chain so that the hanks will all receive the stream of hot air. *C. O. C.*

**Driving Mechanism for Dye Jigs.** F. Roselene. *U.S.P. 2,513,209.*

A simple and effective driving mechanism that does not put unwanted strain on the cloth comprises a pair of rollers which are positively driven from a common power source. *C. O. C.*

**Milling.** E. B. Bates. *B.P. 644,649.*

At each passage through the machine a measurement representative of the length of the fabric is made, milling being stopped when the measurement drops to a predetermined value. Means whereby a length indicator can be incorporated in a milling machine are described. *C. O. C.*

**Applying Colour to Venetian Blind Ladder-tape.** Russell Manufacturing Co. *B.P. 643,873.*

Apparatus is described for printing the outside surface of one of the main tapes one colour and that of the other tape another colour. *C. O. C.*

**Feeding Cloth on to a Pin Stenter.** Ferranti Ltd. and J. H. France. *B.P. 644,523.*

A movable feed wheel conveys the cloth over a first arcuate path. At least part of the periphery of a smaller

drive wheel follows a second arcuate path placed on the concave side of the first arcuate path. Outwardly projecting pins are fastened to the periphery of the drive wheel. Some of these pins contact the convex side of part of the first arcuate path, so that they pierce the cloth and drive it at a velocity approximately equal to the peripheral velocity of the feed wheel. *C. O. C.*

**Continuous Decatizing.** Richmond Piece Dye Works Inc. *B.P. 644,388.*

Results as good as those obtained by batch working are obtained by passing the fabric between a portion of the face of a rotatable perforate hollow suction cylinder and a blanket to hold it in close contact with the cylinder. High-velocity steam orifices extend longitudinally along the cylinder close to the blanket. An arcuate face plate parallel to the cylinder surface surrounds the orifices. *C. O. C.*

**Fabric-folding Machine.** Richmond Piece Dye Works Inc. *B.P. 644,797.*

Cloth is continuously laid in transverse zig-zag folds by a machine in which two or more spaced parallel and horizontal carrier rods move continuously in a closed orbit perpendicularly to their lengths. The cloth is fed to that end of the orbit from which the rods move along the upper part of the orbit, so that it is borne by a rod during passage of the latter along the upper part of its orbit, and laps over and falls vertically from the rod which is at any time at the smallest horizontal distance from the other end of the orbit. *C. O. C.*

**Opening Doubled Cloth.** A. Bieber, L. Bieber, and Express Patent Co. (Liverpool) Ltd. *B.P. 644,657.*

**Continuous Embossing of Artificial Leathers and Similar Materials.** J. K. Mason, P. P. Crisp, and Firestone Tire & Rubber Co. *U.S.P. 2,514,213.*

**Nylon Hose Preboarding Machine.** Paramount Textile Machinery Co. *B.P. 643,641.*

The machine comprises a horizontal insulated retort, and usually two carriages carrying a number of metal hosiery forms. Each carriage is introduced alternately into the retort, entering along parallel tracks. The machine is largely devoid of automatic mechanism, and the movement of the carriages, the closing of the door, and manipulation of the steam inlet and exhaust valves are manual. The door is sealed during operation by the steam pressure inside the chamber. A main carriage carrying the two form carriages can be moved transversely along rails across the mouth of the retort, enabling the carriages to be introduced alternately. The metal forms mounted edgewise in the carriage are horizontally disposed as the carriage enters the retort, but after removal, means are provided for tilting the carriage in order that the hose may be removed, and the forms replenished. *G. E. K.*

**Power-operated Garment Press.** R. G. Whitaker Ltd. and J. R. S. Richardson. *B.P. 643,951.*

A press is described in which the duration of pressing is automatically controlled. *C. O. C.*

**Package Dyeing.** E. A. Stienen. (VIII, p. 43.)

### II—WATER AND EFFLUENTS

**Ion-exchange Characteristics of Chemically Modified Cotton Fabrics.** C. L. Hoffpauir and J. D. Guthrie. *Text. Research J., 20, 617-620 (Sept. 1950).*

The preparation of modified cotton ion-exchangers by treating cotton sheeting with various reagents is described.

Sulphoethyl cellulose, phosphorylated cellulose, carboxymethyl cellulose, cellulose hydrogen succinate, aminoethyl cellulose, diethylaminoethyl cellulose, and quaternary aminated cellulose modifications were examined for their efficiency as ion-exchange bodies. Maximum exchange capacity was shown by phosphorylated cotton cellulose, which appears to behave as a dibasic acid. P. C.

**Textile Industry and Stream Pollution.** J. A. McCarthy. *Amer. Dyestuff Rep.*, 39, P 732-P 735 (30th Oct. 1950).

An account is given of stream pollution by textile effluents, together with the causes and prevention of undesirable effects. J. W. B.

**Wastes Treatment in Cotton Finishing Plants.** G. G. Bogren. *Amer. Dyestuff Rep.*, 39, 669-671 (2nd Oct. 1950).

The nature of cotton-finishing effluents and methods of purification are discussed. Emphasis is laid on economy of operation, such as the use of acid wastes in the neutralization and precipitation of alkaline liquors. J. W. B.

**Recovery of Grease from Wool Scouring Waste and Abatement of Stream Pollution through the Calcium Hypochlorite Process.** A. A. Cameron. *Amer. Dyestuff Rep.*, 39, 667-668 (2nd Oct. 1950).

A non-scientific account is given of a serious problem of effluent purification, and its solution through use of the calcium hypochlorite process. There are some interesting comments on the economic aspect of plant operation and disposal of by-products. J. W. B.

#### PATENT

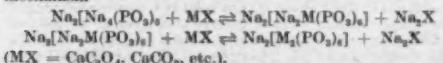
**Softening Water and Removing Carbon Dioxide from it.** C. H. V. Pape. *B.P.* 643,805.

The raw water is passed through a mixture of a cation-exchange material and a compound of a Group II metal having affinity for carbon dioxide, e.g. a mixture of a zeolite and burnt magnesite or calcium carbonate. C. O. C.

## III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

**Hexametaphosphates. I—Preparation and Properties of Sodium Hexametaphosphate and a Study by Conductivity Data of the Extent of Complex Formation with Calcium Ions.** R. C. Mehrotra and N. R. Dhar. *Proc. Natl. Inst. Sci. India*, 16, 59-65 (1950); *Chem. Abs.*, 44, 8278 (25th Sept. 1950).

Pure sodium hexametaphosphate was prepared by heating  $\text{Na}(\text{NH}_4)\text{HPO}_4 \cdot 3\text{H}_2\text{O}$  to 700°C. and rapidly quenching the melt. A 5% aqueous solution forms soluble complexes with salts of Ca, Sr, Ba, Mg, Zn, Mn, Pb, Al, Be, La, Th, Zr, Ce, Fe, and UO<sub>4</sub> by the following mechanism—



**II—Study by Conductivity Measurements of the Complexes formed by Sodium Hexametaphosphate with Strontium, Barium, and Lead Ions.** *Ibid.*, 67-69.

Sr, Ba, and Pb sulphates dissolve in aqueous sodium hexametaphosphate to form  $\text{Na}_2\text{M}(\text{PO}_5)_6$  (M = Sr, Ba, or Pb). Only Pb tended to react a stage further, forming  $\text{Na}_2\text{Pb}_2(\text{PO}_5)_6$ . C. O. C.

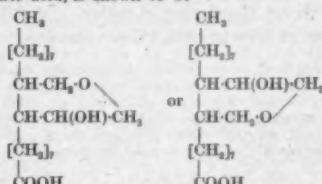
**Chemicals for Textile Treatment [and Dye Intermediates]. II—p-Aminocetylbenzene and Cetyl-*o*-toluidine.** Y. Oshima and M. Hayashi. *J. Soc. Chem. Ind. Japan*, 50, 143, 144 (1947); *Chem. Abs.*, 44, 9154 (10th Oct. 1950).

Aniline condensed with cetyl alcohol using a  $\text{ZnCl}_2$  catalyst yielded *p*-cetyl aniline (I). *o*-Toluidine similarly yielded cetyl-*o*-toluidine (II). On rayon staple II acted as a water-repellent agent. On acetylation I and II yield *p*-acetylaminocetylbenzene and cetyl-2-acetylaminocetylbenzene respectively. On oxidation I yielded *p*-chlorobenzoic acid and *p*-chloroethylbenzene [sic]. Dyes with I and II as the diazo components were prepared. C. O. C.

#### Condensation of Oleic Acid with Formaldehyde.

G. V. Pigulevsky and M. G. Tatarskaya. *J. Gen. Chem. (U.S.S.R.)*, 20, 1456-1467 (Aug. 1950).

The product, of composition  $\text{C}_{21}\text{H}_{40}\text{O}_4$ , which is obtained by the action of formaldehyde on oleic acid in presence of sulphuric acid, is shown to be—



A number of derivatives of this substance and of the keto acid formed by oxidation were prepared and characterized. The unsaturated acid formed by a dehydration reaction involving the secondary alcohol group was isolated, and the products of its ozonization were examined. A. E. S.

**Pioneer Surfactant.** M. L. Kastens and J. J. Ayo, Jr. *Ind. Eng. Chem.*, 42, 1626-1638 (Sept. 1950).

The development of Igepon-type detergents is described and a detailed account of the complete manufacturing process of Igepon T is given. The patent covering the manufacture of Igepon is extremely wide and specifies a compound of the type  $\text{R}^1\text{-CO-NR}^2\text{R}^3$ . In general this is restricted to  $\text{R}^1\text{-CO-NR}^2\text{-CH}_2\text{CH}_2\text{SO}_3\text{R}^4$ , viz. *N*-acyl-*N*-alkyltaurates. This gives a possible number of suitable products of the order of 1000. So far about 200 have been examined, and the properties may be controlled within wide limits. This is discussed from the aspect of chain length, detergency, and availability of the raw materials. The latest development is the use of these Igepons in the production of specialist types accentuating maximum efficiency in one direction as distinct from a universal "soap". W. J. M.

**Use of Chlorite in the Textile Industry.** H. J. Meybeck. *Textil-Rund.*, 5, 349-362 (Sept. 1950).

Sodium chlorite is marketed as the trihydrate or in the anhydrous form, the latter being more stable in storage. The properties of sodium chlorite, the effect of pH on the stability of aqueous solutions, and the reactions which take place between sodium chlorite and hypochlorite, hydrogen peroxide, and aldehydes are described. Details are given of the effect of sodium chlorite on metals, alloys, plastics, and other types of coatings applied to vessels used in the textile trade. The use of sodium chlorite in bleaching cotton, viscose rayon, flax, hemp, jute, acetate rayon, and polyamide fibres is described, and found to be of wide application. Chlorite has limited use on protein fibres such as wool, as the fibre tends to be attacked.

B. K.

#### PATENTS

**Detergents.** California Research Corp. *B.P.* 645,129. Phenyl-substituted alkanes in which the alkane portion of the molecule contains 12-15 C and their sulphonated derivatives have excellent detergent properties. C. O. C.

**Rendering Wool Textiles Shrink-resistant with Butadiene Copolymers.** J. B. Rust, Montclair Research Corp., and Ellis-Foster Co. *U.S.P.* 2,514,517. Copolymers of 1,3-butadiene and a vinyl ester which polymerizes to a hard resinous material give an excellent shrink-resistant finish to wool textiles. C. O. C.

**Nitrodithioacetates as Fungicides.** E. E. Gilbert and Allied Chemical & Dye Corp. *U.S.P.* 2,513,018. Metal salts of compounds of formula  $\text{NO}_2\text{-CR}^1\text{R}^2\text{-CSH}$  ( $\text{R}^1 = \text{H}$  or  $\text{CH}_3$ ,  $\text{R}^2 = \text{H}$ ,  $\text{CH}_3$ , or  $\text{C}_2\text{H}_5$ , the total number of carbon atoms in the compound being  $< 5$ ), e.g. copper nitrodithioacetate, have good fungicidal properties. C. O. C.

**Rosin-ammonium Phenoxides as Fungicides.** R. Rosher and Hercules Powder Co. *U.S.P.* 2,513,429. Rosin-ammonium phenoxides obtained from rosin amines and phenols have fungicidal properties much greater than those of either the phenol or the rosin amine from which they are prepared. They are non-volatile, do not bloom, and are insoluble in water. C. O. C.

**Vulcanization Accelerators and Agents for improving the Adhesion of Rubber to Textiles.** Monsanto Chemical Co. *B.P.* 643,688.

The products obtained by treating mercaptothiazoles with formaldehyde and ammonia in aqueous medium are useful as vulcanization accelerators and for treating tyre cord to improve its adhesion to rubber. C. O. C.

**Emulsions of Stearic Acid.** Nopco Chemical Co. *B.P.* 643,765.

Saturated fatty acids of 12-24 C can be made into aqueous dispersions of high stability to variations in pH, to electrolytes, and to dilution with water, by using as the dispersing agent an ester of a fatty acid with a polyethylene glycol of mol. wt. 600-4000. Dispersions of commercial stearic acid made in this way are very suitable for paper coating. C. O. C.

**Tanning Agents.** L. Bolgar. (XII, p. 47.)

## IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

**Chemical Engineering Unit Processes Review.** *Ind. Eng. Chem.*, 42, 1639-1768 (Sept. 1950).

This covers kinetics and equilibria, alkylation, amination by reduction, ammonolysis, esterification, fermentation, Friedel-Crafts reactions, halogenation, hydration and hydrolysis, hydrogenation and hydrogenolysis, isomerization, nitration, polymerization, pyrolysis of coal and shale, pyrolytic and catalytic decomposition of hydrocarbons, sulphonation, and other unit processes. C. J. W. H.

**Hydrazine in Organic Chemistry.** G. D. Byrkit and G. A. Michalek. *Ind. Eng. Chem.*, 42, 1862-1875 (Sept. 1950).

Anhydrous hydrazine is more reactive than hydrated hydrazine and is now commercially available. Their reactions and those of the hydrazine salts and their derivatives are reviewed and a very extensive bibliography (384 references) is given. The derivatives include the following substituted hydrazines—heterocyclic hydrazines, hydrazine acids and esters, hydrazones, azines, hydrazides and their polycondensation products, azides, semicarbazide and thiosemicarbazide, semicarbazones and thiosemicarbazones, aminoguanidine, and heterocyclic rings produced by hydrazine synthesis. Possible uses are described in a variety of fields—organic intermediates, pharmaceuticals, dyes, resins, photographic developers, explosives, and other similar types of fields. The toxicity of anhydrous hydrazine and precautions required when handling it are discussed. It is suggested that where the hydrate or aqueous solutions may be used it is preferable to do so, as they are much safer to handle. W. J. M.

**Kinetics and Mechanism of Aromatic Nitration—VI-VIII.** *J.C.S.*, 2628-2684 (Oct. 1950).

**VI—Nitration of Phenols and Phenolic Ethers: the Concomitant Dealkylation of Phenolic Ethers. The Rôle of Nitrous Acid.** C. A. Bunton, E. D. Hughes, C. K. Ingold, D. I. H. Jacobs, M. H. Jones, G. J. Minkoff, and R. I. Reed. *Ibid.*, 2628-2656.

**VII—Products of Nitration of Aniline Derivatives, especially of Dimethylaniline. The Concomitant Dealkylation of Dialkylanilines.** J. Glazer, E. D. Hughes, C. K. Ingold, A. T. James, G. T. Jones, and E. Roberts. *Ibid.*, 2657-2678.

**VIII—Rearrangement of Aromatic N-Nitroamines.** E. D. Hughes and G. T. Jones. *Ibid.*, 2678-2684.

**Hydrogen Fluoride-Boron Trifluoride Extraction of Xylene Isomers.** D. A. McCaulay, B. H. Shoemaker, and A. P. Lien. *Ind. Eng. Chem.*, 42, 2103-2107 (Oct. 1950).

*m*-Xylene may be selectively removed from its isomers by extraction with a hydrogen fluoride-boron trifluoride ( $\text{HF-BF}_3$ ) mixture. The extracted *m*-xylene is recovered from the extract solution by vaporizing the  $\text{HF-BF}_3$  at moderately low temperatures. Vapour pressure measurements show that all the isomers react rapidly with  $\text{HF-BF}_3$  to form complexes which are soluble in excess  $\text{HF}$ . A. S. F.

**Reactivity of  $\beta$ -Naphthols and  $\beta$ -Naphthylamines.**

F. Bell and W. H. Hunter. *J.C.S.*, 2903 (Oct. 1950). Experiments to indicate the low reactivity of the 3-position in  $\beta$ -naphthols and  $\beta$ -naphthylamines are described, viz.—Reimer-Tiemann reaction with 1-methyl-2-naphthol gave 2-keto-1-methyl-1-dichloromethyl-1,2-dihydronaphthalene, and an analogous compound with 1-ethyl-2-naphthol. 1-Methyl-2-naphthol would not couple with diazotized picramide. The nitration of 1-bromo-2-toluene-*p*-sulphonamidonaphthalene in pyridine gave the 6-nitro derivative. Attempts to cause migration of the nitroso group in 1-bromo-*N*-nitroso-2-N-methylnaphthylamine failed. Chlorination of *a*-nitroso- $\beta$ -naphthol (cf. Marschalk, *Bull. Soc. chim.*, 43, 1361 (1928)) in the 3-position is confirmed, and is one of the few examples of group entry in position 3. Bromination of 1-bromo-2-naphthol in pyridine, however, led only to the isolation of 6-bromo-1-bromopyridino-2-naphthol.

H. H. H.

**Cyanoanthracenes.** H. Waldmann and R. Streng. *Chem. Ber.*, 83, 167-170 (1950); *Chem. Abs.*, 44, 7300 (25th Aug. 1950).

The preparation of a number of cyanoanthracenes, by dehydrating the corresponding amides by heating with phthalic anhydride, is described. Thus, anthracene-1,5-dicarboxylic acid is refluxed with thionyl chloride in monochlorobenzene, and the resulting acid chloride converted to the 1,5-diamide by warming with aq. ammonia. The 1,5-dicyano compound is obtained by heating this diamide with phthalic anhydride until water stops distilling off. R. K. F.

**Kinetics and Mechanism of Diazotization.** E. D. Hughes, C. K. Ingold, and J. H. Ridd. *Nature*, 166, 642-643 (14th Oct. 1950).

Rates of diazotization of various amines, e.g. aniline and *o*-, *m*-, and *p*-toluidine, have been measured in weakly acidic aqueous solutions of nitrous acid, and in the presence of perchloric acid as the only strong acid, to avoid possible complications introduced by  $\text{HCl}$  or  $\text{HBr}$  (the usual acids employed), such as the formation of  $\text{NOCl}$  or  $\text{NOBr}$ . The true expression for low acidities (i.e.  $< 0.002 \text{ N}$ ) is—

$$\text{Rate} \propto [\text{HNO}_3]^2$$

and is then independent of the nature and concentration of the amine. The conclusion is drawn that  $\text{N}_2\text{O}_3$  is the active agent. At quite low acidities, the concentrations of free amine are large enough to secure that the  $\text{N}_2\text{O}_3$  is destroyed as fast as it is formed, while at high acidities there is so little free amine that the  $\text{N}_2\text{O}_3$  is able closely to approach its equilibrium concentration. H. H. H.

**Diazotization of *p*-Aminoazo Dyes.** S. F. Filippichev. *Doklady Akad. Nauk S.S.R.*, 73, 731-734 (1st Aug. 1950).

The hypothesis, that the inability of *o*-aminoazo dyes to undergo diazotization is due to their existing exclusively in the hydrazone form, is extended to a number of *p*-aminoazo dyes which do not readily diazotize by normal methods. In many cases the dye is a mixture of the two tautomers, and isomerization does not readily occur under the conditions of diazotization; hence only partial conversion into diazonium salt occurs. In other cases the azo tautomer may be present in negligible amount, and diazotization does not occur. However, the azo tautomer may usually be prepared, e.g. by salting out the dye from weakly alkaline soln. or by some more drastic alkaline treatment, and it can then be diazotized completely using the inverted method (addition, in admixture with sodium nitrite soln., to a soln. of hydrochloric acid), but not by the direct method, in which acid is added before nitrite. The pure hydrazone form may be obtained by heating the azo form at 90°C. with hydrochloric acid for 2 hr. It is readily reconverted into the azo form by treating with weak alkali in the cold. The following dyes were prepared both in the pure hydrazone form (blue-violet) and in the azo form (orange)—

- Metanilic acid  $\rightarrow$  *a*-Naphthylamine
- Sulphanilic acid  $\rightarrow$  *a*-Naphthylamine
- 4-Chloroaniline-2-sulphonic acid  $\rightarrow$  *a*-Naphthylamine
- Aniline  $\rightarrow$  1-Aminonaphthalene-7-sulphonic acid
- *p*-Nitroaniline  $\rightarrow$  1-Aminonaphthalene-7-sulphonic acid
- *m*-Nitroaniline  $\rightarrow$  1-Aminonaphthalene-7-sulphonic acid
- Metanilic acid  $\rightarrow$  1-Aminonaphthalene-7-sulphonic acid

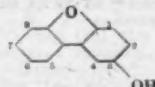
In all cases the hydrazone form was unaffected by an 8-hr. treatment with nitrous acid. A number of other, more readily diazotizable dyes were also examined. A. E. S.

**Metallic Complex Derivatives of Azo Dyes.** R. Specklin. *Teintex*, 15, 451–469 (Oct. 1950).

A review with 62 references to the literature of the chemistry of metallic derivatives of azo dyes, including those Cu, Ni, and Cr compounds which are of interest in dyeing. Benzo Fast Copper (IG), Chlorantine Fast (Ciba), Palatine Fast (IG), and Coprantine (Ciba) dyes are included. B. K.

**Diphenylene Oxide Derivatives.** K. Schimmelschmidt. *Annalen*, 566, 184–206 (1950); *Chem. Abs.*, 44, 5863 (10th Oct. 1950).

The preparation of a wide range of substituted diphenylene oxides is described. Aryl derivatives of 1:4-benzoquinone and quinol were first prepared. Thus quinol was warmed with aqueous potassium perbromate and sulphuric acid, and a solution of diazotized *p*-chloroaniline added after cooling and neutralizing. The resulting 2-*o*-chlorophenyl-1:4-benzoquinone was reduced to the corresponding quinol with hydrosulphite, and heated at 190°C. with 50% KOH to give—



Amino derivatives are included, and their properties compared; e.g. the 4-amino compound couples with diazotized amines (a red dye is produced with diazotized *p*-nitroaniline), whereas the corresponding 2-derivative does not.

R. K. F.

**Formation of Azomethin Dyes in Colour Development.** G. I. Arbusov. *J. Appl. Chem. (U.S.S.R.)*, 23, 886–888 (Aug. 1950).

Previous work (Porai-Koshits, *et al.* *Chem. Abs.*, 40, 1745 (1946)) has shown that the dye formed by the action of freshly precipitated silver bromide on a mixture of a *p*-aminodialkylaniline and 1-phenyl-3-methyl-5-pyrazolone (or an *o*-naphthol deriv.) is identical with that produced by the condensation of *p*-nitrosodialkylaniline and the same component. It was therefore surmised that in the photographic colour development process, using *p*-aminodialkylaniline as developer and the same second component as before, the same dyes are formed. This is now confirmed in the case of the dye formed in the photographic emulsion from *p*-aminodimethylaniline and 1-phenyl-3-methyl-5-pyrazolone. The dye was extracted from the emulsion, and its absorption spectrum was determined and shown to be identical with that of the azomethin dye obtained from the same components and silver bromide, or from *p*-nitrosodimethylaniline and 1-phenyl-3-methyl-5-pyrazolone. A. E. S.

**Indophenol—II.** E. Ziegler, K. Gartler, and E. Wiesnerberger. *Monatsh.*, 80, 759–764 (1949); *Chem. Abs.*, 44, 7803 (10th Sept. 1950).

Examples are given to show that formation of indophenols *p*-HO-C<sub>6</sub>H<sub>4</sub>N<sub>2</sub>C<sub>6</sub>H<sub>5</sub>O is not dependent upon an open position *para* to the HO group; e.g. indophenol can be obtained from *pp'*-dihydroxydiphenylmethane and *p*-quinoneimine chloride by cationoid substitution followed by elimination of the substituent *para* to the original OH group. C. O. C.

**Chemical Properties of Sulphur Black T.** H. Hiayama. *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 51, 92–94 (1948); *Chem. Abs.*, 44, 9153 (10th Oct. 1950).

By desulphurizing and sulphurizing Sulphur Black T prepared under various conditions, its characteristic properties as a sulphur dye become apparent when the C:S ratio is > 24:5. Desulphurizing N<sub>2</sub>-dyes by boiling with Na<sub>2</sub>S yields N<sub>2</sub>-dyes. Products dried in a stream of N<sub>2</sub> at 100°C. contain 3 mol. of H<sub>2</sub>O and form a tri-sodium salt. Deterioration (due to oxidation) can be avoided by keeping the dyes in a vacuum. C. O. C.

**Combining Positions of Sulphur in Sulphur Black T.** H. Hiayama. *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 51, 94–95 (1948); *Chem. Abs.*, 44, 9153 (10th Oct. 1950).

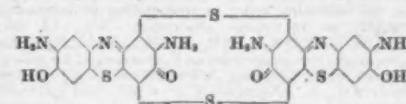
*o*-, *m*-, and *p*-Nitrophenol, *p*-aminophenol, indophenol, nitroso-*o*-cresol, nitroso-*m*-cresol, 2-chloro-4:6-dinitro-

phenol, 4-chloro-2-nitrophenol, 2:6-dichloro-4-nitrophenol, 2:4-dichloro-6-nitrophenol, and picric acid were all sulphurized, and the structures of the products determined. The effects of Cl atoms and position and number of NO or CH<sub>3</sub> groups on the properties of the dyes were investigated. C. O. C.

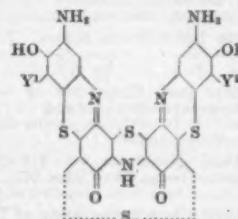
**Chemical Structure of Sulphur Black T.** H. Hiayama.

*J. Chem. Soc. Japan, Ind. Chem. Sect.*, 51, 97–98 (1948); *Chem. Abs.*, 44, 9154 (10th Oct. 1950).

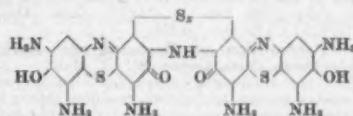
Sulphur Black T is believed to have the constitution—



(x = 2–7) when the sulphurizing temp. is 103–105°C., and—



(Y = H; x = 2–7) when the sulphurizing temp. is > 110°C., above which S enters the molecule at positions Y<sup>1</sup> and Y<sup>2</sup>. The sulphur dye from picric acid has the constitution—



(x = 2–7), some of the amino groups in this molecule combining with other molecules of the dye to form -NH-bridges. C. O. C.

**Dye from Naphthoeresorcinol and Glucuronic Acid.**

A. Ogata and Y. Nozaki. *J. Pharm. Soc. Japan*, 63, 416–425 (1943); *Chem. Abs.*, 44, 7242 (25th Aug. 1950).

The purple dye formed in the Tollens naphthoeresorcinol reaction used in the detection and determination of glucuronic acid was prepared in a pure state by carrying out the reaction in glacial acetic acid in presence of zinc chloride, conc. sulphuric acid, or oxalic acid. It was found to be a hydroxydinaphthylfurylmethane derivative. R. K. F.

**Colour of Organic Dyes and the Planarity of their Molecules.** A. I. Kiprianov and I. K. Ushenko.

*Izvestiya Akad. Nauk S.S.R., Otdel. Khim. Nauk*, 492–500 (Sept.–Oct. 1950).

A review is made of recent work, some not previously published, by the authors and their associates on the influence of the degree of planarity of a dye molecule on its properties (cf. *J.S.D.C.*, 66, 450 (Aug. 1950)). The dyes considered are of the polymethin, triphenylmethane, and azo groups. It is shown that the introduction of bulky substituents, which by their steric effects disturb the planarity of the dye molecule, results in a reduction in colour intensity and a displacement, in one direction or the other, of the absorption bands. These effects are accompanied by a greater sensitivity of the dye to acids and alkalies, and by a decreased fastness to light. In addition to considerations of the coplanarity of aromatic and/or heterocyclic nuclei amongst themselves, particular consideration is given to the disturbance of the coplanarity between a nucleus and an attached dimethylamino group. A. E. S.

**Investigations in the Field of Polycyclic Compounds.**

**XI—Effect of the Nature of the Substituent on the Colour of 4-Substituted 1-Aminoanthraquinones. XII—Effect of the Nature of the Substituent on the Colour of 4-Substituted 1-Benzoylaminanthraquinones.** A. M. Lukin and K. K. Mozgova. *J. Gen. Chem. (U.S.S.R.)*, 20, 1504-1509 and 1510-1513 (Aug. 1950).

The absorption spectra were determined in the visible region for solutions of 4-substituted 1-aminoanthraquinones in ethanol and in chlorobenzene. The substituents fall in the following order, corresponding to increasing wavelength at maximum absorption— $\text{NO}_2$ ,  $\text{Cl}$ ,  $\text{Br}$ ,  $\text{OCH}_3$ ,  $\text{OH}$ ,  $\text{NH-C}_6\text{H}_4\text{NH}_2$ ,  $\text{N}(\text{CH}_3)_2$ ,  $\text{NH-C}_6\text{H}_5$ . This is, in the main, the same order as that observed for mono-substituted anthraquinones. When the second substituent is  $\text{OH}$  or  $\text{NH}_2$ , or substituted  $\text{OH}$  or  $\text{NH}_2$ , the spectrum has a second maximum at a shorter wavelength. Similar determinations were made for solutions of 4-substituted 1-benzoylaminanthraquinones in chlorobenzene. The position of the absorption maximum varied with the substituent in exactly the same way as before. A. E. S.

**Vat Dyes of the Indanthrone Series.** T. Maki and T. Mine. *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 51, 13-16 (1948); *Chem. Abs.*, 44, 9151 (10th Oct. 1950).

**XV—Preparation and Properties of 1-Amino-2-bromo-4-hydroxyanthraquinone.**

The method of *G.P.* 203,083, has, despite many difficulties, been successfully applied.

**XVI—Synthesised Reactions of 4:4'-Dihydroxyanthraquinone (Indanthren Blue 5G).**

Various derivatives have been prepared and described.

**XVII—Constitution and Preparation of Indanthren Blue 3G.**

Indanthren Blue 3G, usually regarded as hydroxyanthraquinone, probably contains indanthrone and indanthrone-sulphonic acid. C. O. C.

**Vat Dyes of the Benzanthrone Series.** *J. Soc. Chem. Ind. Japan*, 50, 140-143 (1947); *Chem. Abs.*, 44, 9151-9152 (10th Oct. 1950).

**XXVI—Preparation of Benzanthrone-Acridone (Indanthren Olive Green B).** T. Maki.

A high yield was obtained by reducing 3-benzanthronyl-1-aminoanthraquinone to Indanthren Olive Green B by heating the intermediate compound at 100°C. with alcoholic KOH or NaOH.

**XXVII—Constitution and Synthesis of Indanthren Navy Blue BRF.** T. Maki and A. Kikuchi.

Examination of the I.G. product gave its main components as 6-bromovianthrone and 6:6':6-dibromovianthrone with a small amount of 6:6':8-tribromovianthrone. C. O. C.

**XXVIII—Synthesis of Derivatives of Benzanthrone-pyrazole and Constitution of Indanthren Grey**

M. T. Maki.

Indanthren Grey M is a mixture of 57 parts of benzanthrone-pyrazolemonooanthraquinonylimide, 43 parts of dianthaquinonyldiimide, and 4 parts of benzanthrone-acridone. C. O. C.

**New Synthesis of Indigo.** J. Harley-Mason. *J.C.S.*, 2907 (Oct. 1950).

2-Nitro-1-*o*-nitrophenylethyl alcohol is converted into indigo in high yield by treatment with alkaline sodium dithionite (hydrosulphite), or in lower yield by other alkaline reducing agents, including ammonium sulphide, zinc dust and ammonium chloride, and ferrous sulphate and sodium hydroxide (cf. Thiele, *Ber.*, 32, 1293 (1899)).

H. H. H.

**Fluorescence of Magnesium Phthalocyanine and of Chlorophyll under Various Conditions—Change in the Electronic-Vibrational Structure of the Fluorescence Spectrum of Magnesium Phthalocyanine on Forming Complexes with Other Molecules in the Adsorbed State.** V. F. Gachkovsky. *Doklady Akad. Nauk S.S.R.*, 73, 963-966 (11th Aug. 1950).

Following the work previously reported (cf. *J.S.D.C.*, 66, 598 (Nov. 1950)), in which the fluorescence of magnesium phthalocyanine adsorbed on magnesium oxide from soin. was shown to depend on the nature of the solvent and its impurities, experiments have now been

carried out on adsorption from the gas phase at low pressure, all impurities being rigorously excluded. The adsorbate did not fluoresce when exposed to ultra-violet radiation. Various pure vapours and gases were admitted to the adsorbate under low pressure. The vapours of benzene, toluene, and tetrahydronaphthalene did not promote fluorescence, but the admittance of  $\text{O}_2$ ,  $\text{Cl}_2$ ,  $\text{CO}_2$ ,  $\text{NH}_3$  or  $\text{H}_2\text{S}$ , or the vapour of ethanol, water, diethyl ether, dimethyl ether, phenol, or iodine, resulted in the appearance of a fluorescence spectrum with a principal maximum at about 700 m $\mu$ . and a series of subsidiary maxima of continually diminishing intensity extending into the longer waves. The positions of the maxima depend on the nature of the substances admitted, and, in particular, the spacing of the subsidiary maxima depends on the mol. wt. of the substance admitted. The spectra are to be interpreted as due to electronic transitions from the excited level to the various vibration states of the normal level. It is indicated how these results and those of other workers are interpreted in terms of the theory of complex formation at the central Mg, which was advanced in the previous communication.

A. E. S.

**Porphyrin Colouring Matters and Porphyrin-Metallic Complexes in Swiss Bitumens. Geochemical Researches—V.** M. Blumer. *Helv. Chim. Acta*, 33, 1627-1637 (Oct. 1950).

A series of Swiss bitumens have been qualitatively examined by means of their absorption spectra for the presence of porphyrin dyes and metallic complexes. Hydrochloric acid fractionation from isopropyl ether solution has enabled partial separation of the dyes. The content of vanadium porphyrin complex amounts to ~0.38%, with ~0.004% of free porphyrin, and traces of green pigment. Possible causes of complex formation are discussed.

H. H. H.

**Carotenoids and Vitamin A.** R. F. Hunter. *Research*, 3, 453-461 (Oct. 1950).

A review with 61 references. C. O. C.

**Complete Synthesis of  $\beta$ -Carotene.** H. H. Inhoffen, F. Bohlmann, K. Bartram, and H. Pommer. *Chem. Ztg.*, 74, 285 (1950).

**Synthesis of Carotenoids. IV—Synthesis of an  $\epsilon_1$ -Carotene.** P. Karrer and C. H. Eugster. *Helv. Chim. Acta*, 33, 1433-1437 (Oct. 1950).

**Synthesis of Carotenoids. V—Simultaneous Synthetic Formation of  $\epsilon_1$ -Carotene,  $\beta$ -Carotene, and DL- $\alpha$ -Carotene.** P. Karrer and C. H. Eugster. *Helv. Chim. Acta*, 33, 1952-1954 (Oct. 1950).

**Fagopyrin, a Photodynamic Pigment from Buckwheat (*Fagopyrum esculentum*).** H. Brockmann, E. Weber, and E. Sander. *Naturwissenschaften*, 37, 43 (1950).

**Plant Pigments. Rutin from Flower of *Magnolia kobus*.** K. Hayashi and K. Ouchi. *Misc. Rept. Research Inst. Nat. Resources (Japan)*, No. 14, 1-4 (1949).

**Pigment of Purple Rice.** K. Hayashi. *Misc. Rept. Research Inst. Nat. Resources (Japan)*, No. 9, 1-8 (1946); *Chem. Abs.*, 44, 9013 (10th Oct. 1950).

The purple pigment which develops particularly well on the reverse side of the leaves of *Oryza sativa* var. *atropurpurea* well exposed to the sun is cyanidin monoglucoside. C. O. C.

**Plant Pigments. I—Yellow Colouring Matter from the Flower of *Meratia praecox*, especially its New Flavone Glucoside.** K. Hayashi and K. Ouchi. *Misc. Rept. Research Inst. Nat. Resources (Japan)*, No. 11, 41-44 (1948); *Chem. Abs.*, 44, 9013 (10th Oct. 1950).

The yellow pigment is quercetol-3-diglucoside and has been named *meratin*.

**II—Colouring Principle of a Japanese Vegetable Dye from "Kariyasu" Grass.** K. Hayashi and T. Inoue. *Ibid.*, No. 12, 11-13 (1948).

The pigment present in the grass *Arthraxon hispidus* Makino var. *brevistylus* Hara is the monoarabinoside of a flavone, probably luteolin or a derivative of it. C. O. C.

**Pigment Systems of Purple Bacteria.** L. K. Osmitkayeva. *Mikrobiologiya*, 19, 152-170 (1950); *Chem. Abs.*, 44, 8424 (25th Sept. 1950).

A review of red, green, and yellow pigments in *Bact.*

*rubescens*, the Thiorhodaceae, and the Athiorhodaceae as identified by chemical and spectroscopic analysis. There are 85 references.

C. O. C.

**Carbon Black Manufacture in Great Britain.** F. H. Amon. *Trans. Inst. Rubber Ind.*, 26, 177-191 (Oct. 1950).

The economic and technological factors which have influenced the manufacture of carbon black in the U.S.A. are discussed in relation to the impending manufacture of fully reinforcing carbon black in Great Britain. The development of the furnace process and the increasing use of liquid hydrocarbons in place of gaseous hydrocarbons are described. Furnace oil black of the HAF type is compared with channel black.

C. O. C.

## PATENTS

**Azoic Dyes — Sulphomethylidicyandiamide as Stabilizer.** J. M. Smith, Jr., and American Cyanamid Co. *U.S.P.* 2,502,897.

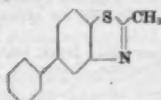
Sulphomethylidicyandiamide, formed by warming dicyandiamide with aqueous formaldehyde-alkali bisulphite solution, has the probable constitution—



Its soluble alkali-metal salts react with diazo compounds to give stable, soluble triazenes, which regenerate the diazo compound on acid steaming; hence they may be used in the production of azoic prints. E. S.

**2-Methyl-5-phenylbenzothiazole and Quaternary Salts thereof.** G. V. Zandt, L. G. S. Brooker, and Eastman Kodak Co. *U.S.P.* 2,515,913.

## 2-Methyl-5-phenylbenzothiazole—



is obtained by reducing bis-3-nitro-4-diphenyl disulphide with Zn and acetic acid and acetylating the reduction product with acetic anhydride or acetyl chloride. Its quaternary salts are intermediates for methin dyes.

C. O. C.

**Cyanoacetamides as Azo Coupling Components in Diazoic Layers.** General Aniline & Film Corp., W. H. von Glahn, and L. N. Stanley. *B.P.* 644,186.

Compounds of formula  $\text{CN}-\text{CH}_2-\text{CO}-\text{R}$  ( $\text{R}$  = amino, aralkylamino,  $\alpha$ -aminodihydroheterocyclic, ureido, thioureido, guanidino, cyanoamino, or carbalkoxyamino (urethano)) can be used either alone or as shading agents for other azo coupling components to give images of excellent covering power and opacity to ultra-violet radiation.

C. O. C.

**Photographs in Blue-Black Tones.** General Aniline & Film Corp., F. Dersch, and N. Heimbach. *B.P.* 644,788.

Compounds of formula  $\text{R}^1-\text{CO}-\text{NH}-\text{CS}-\text{Y}-\text{R}^2$  or—



( $\text{R}^1$  and  $\text{R}^2$  = independent similar or dissimilar Alk;  $\text{Y} = \text{O}$ ,  $\text{S}$ , or  $\text{Se}$ ;  $\text{Z} = -\text{CR}^3\text{R}^4$  or  $-\text{CR}^3\text{R}^4\text{CR}^5\text{R}^6$ ;  $\text{R}^3$  and  $\text{R}^4$  = Alk;  $\text{R}^5$ ,  $\text{R}^6$ ,  $\text{R}^7$ , and  $\text{R}^8$  =  $\text{H}$  or Alk), e.g. 2-mercaptop-4-keto-5:5-dimethylthiazolidine, are blue-black toning agents for use in photographic emulsions or developers.

C. O. C.

**Polyvinylacetol Colour Formers.** E. L. Martin and du Pont. *U.S.P.* 2,513,189.

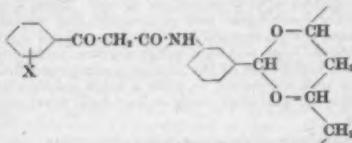
Polyvinylacetol colour formers are obtained in increased yield and free from by-products by condensing a monomeric colour-forming aldehyde or lower acetal thereof with a synthetic hydroxyl polymer containing recurring linear vinyl alcohol  $-\text{CH}_2\text{CH}(\text{OH})-$  groups.

C. O. C.

**Acetals of Polyvinyl Alcohol and m-Benzoylacetal-amidobenzaldehydes—Uye Intermediates.** D. M. McQueen and du Pont. *U.S.P.* 2,513,190.

Treating a vinyl alcohol polymer containing many recurring intralinear  $-\text{CH}_2\text{CH}(\text{OH})-$  groups with a

*m*-benzoylacetalamidobenzaldehyde (or a lower acetal thereof with an alkanol of 1-4 C or a 1:2- or 1:3-alkanediol or glycol of 2-4 C) yields acetals containing colour-former units of structure—

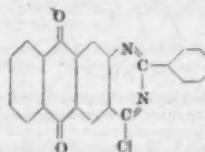


( $\text{X} = \text{H}$ ,  $\text{Cl}$ ,  $\text{Br}$ ,  $\text{CH}_3$ ,  $\text{O}-\text{CH}_3$ ,  $\text{COOC}_2\text{H}_5$ , or  $\text{COOH}$ ). They are intermediates for azomethine dyes.

C. O. C.

**Quinazoline Derivatives.** Ciba Ltd. *B.P.* 642,129.

An acylaminanthraquinone-*o*-nitrile, prepared by treating the corresponding acylamino-*o*-halogeno compound with cuprous cyanide, is heated with phosphorus pentachloride to give a phthaloyl-quinazoline of use as a dye intermediate. Thus the compound—



is made by heating 3-cyano-2-benzoylaminanthraquinone and phosphorus pentachloride in nitrobenzene at 180-185°C.

R. K. P.

**Brown, Chrome Monoazo Dyes.** Ciba Ltd. *B.P.* 643,354.

Metallizable *oo'-*dihydroxymonoazo dyes are made by coupling a diazotized 6-acylamino-4-nitro-2-aminophenol with a *p*-alkylphenol. Alternatively 4:6-dinitro-2-aminophenol may be used as the diazotized amine, the 6-nitro group being reduced after coupling, and then acylated. Thus diazotized 6-acetylamino-4-nitro-2-aminophenol is coupled with *p*-cresol in presence of caustic soda and pyridine to yield a dark brown dye applicable to wool by either the chrome mordant or the metachrome process.

E. S.

**Chromiferous Monoazo Dye.** D. E. Kvalnes, B. C. Carson, and du Pont. *U.S.P.* 2,508,404.

The monoazo compound 4-chloro-2-aminophenol-6-sulphonic acid- $\rightarrow$ 1:3-dihydroxy-*o*-quinolone can be successfully converted to a chromium complex containing 1 atom of Cr per mol. of monoazo compound by heating with aqueous chromium fluoride or chromium acetate at 95-125°C., providing that the concentration of other salts present does not exceed 1% NaCl or its equivalent. The product dyes wool and nylon bright bluish pink from an acid bath.

E. S.

**Maroon Azoic Pigment for Alkyd Resin Enamels.** J. H. Cooper and du Pont. *U.S.P.* 2,508,447.

The maroon azoic pigment 5-nitro-2-aminonaphtho-2-hydroxy-3-naphthoic *o*-toluidide is similar in tone to 3-nitro-4-aminotoluene- $\rightarrow$ 2-hydroxy-3-naphthoic *m*-nitroanilide, but has much superior durability when incorporated in alkyd resin enamels.

A. A. Brizzolara and du Pont. *U.S.P.* 2,508,448.

Coating compositions containing 5-nitro-2-aminonaphtho-2-hydroxy-3-naphthoic *o*-toluidide become viscous on keeping. This can be overcome by replacing 5-25% of this pigment with the pigment 5-nitro-2-aminonaphtho-2-hydroxy-3-naphthoic acid or a metallic (especially manganese) salt thereof.

E. S.

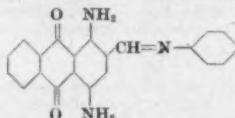
**Metallizable, Substantive, Tetrakisazo Dyes.** Ciba Ltd. *B.P.* 643,535.

Tetrakisazo dyes which may be treated with metallic (especially copper) salts in substance or on the fibre are made by coupling 2-amino-5-naphthol-7-sulphonic acid first under acid conditions with a diazotized amminon compound  $\text{R}^1\text{N}^+\text{N}^-\text{R}^2\text{NH}_2$  ( $\text{R}^1$  = a benzene residue containing OH and COOH *ortho* to one another;  $\text{R}^2$  = a benzene residue in which the  $\text{NH}_2$  is *para* to the azo group).

and then under alkaline conditions with a diazotized aminoazo compound  $R^2-N=N-R^4-NH_2$ , ( $R^2$  = a benzene residue containing OH and COOH *ortho* to one another;  $R^4$  = a benzene residue in which the NH<sub>2</sub> is *para* to the azo group and *ortho* to an alkoxy group). Thus, 2-amino-5-naphthol-7-sulphonic acid is coupled first under acid conditions with diazotized 4-aminoazobenzene-4'-hydroxy-3'-carboxylic acid, and then under alkaline conditions with the diazo compound of the monoazo compound 5-amino-salicylic acid  $\rightarrow$  cresidine, to yield a black substantive dye applicable by the single-bath or two-bath aftercoppering process.

**1: 4-Diaminoanthraquinone-2-azomethins—Dyes for Cellulose Esters and Superpolyamides.** Ciba Ltd. B.P. 642,592.

1:4-Diaminoanthraquinones containing an azomethin link in the 2-position are blue dyes for cellulose acetate and nylon. They are prepared, e.g. by condensing the corresponding 2-methyl-2-alkaldehyde derivative with an aliphatic or aromatic amine. Thus the compound—

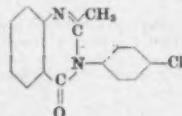


is made by refluxing 1:4-diamino-2-methylanthraquinone, aniline, and potassium carbonate in nitrobenzene for 3 hr.

R. K. F.

**Quinazolones.** A. Guenther, J. F. Morgan, and General Aniline & Film Corp. B.P. 642,134.

An *N*-acyl-*N*-amino cyclic carboxylic acid is condensed with a primary aliphatic or aromatic amine in presence of  $PCl_3$  or  $POCl_3$  to give a quinazolone. Thus 2-methyl-3-*p*-chlorophenyl-4-quinazolone—

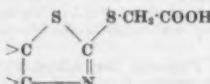


is made by adding  $PCl_3$  to a suspension of *p*-chloroaniline and *N*-acetylanthranilic acid in toluene, and then refluxing for 1-2 hr. The toluene is removed by steam-distilling after making alkaline with  $Na_2CO_3$ , and the product filtered off.

R. K. F.

**Dyes and Pigments from S-2-Thiazolylthioglycolic Acids.** G. Duffin, J. D. Kendall, and Ilford Ltd. B.P. 643,681.

Water-insoluble dyes which cannot be reduced to leuco compounds are obtained by treating a compound of formula—

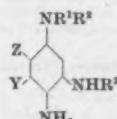


with a monocarboxylic anhydride.

C. O. C.

**Benzopyrazines and Benzopyrazines having fused to the Pyrazine Ring a Benzene, Naphthalene, or Quinoline Ring System.** General Aniline & Film Corp., W. A. Schmidt, and V. Tulagin. B.P. 644,863.

Development of a photographic silver emulsion in presence of a conventional colour former with a compound of formula—



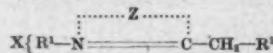
( $R^1$  = H or an aliphatic radical;  $R^2$  = H, Alk, or an aromatic radical;  $R^3$  = an aliphatic or aromatic radical, in the former case linked to the N by a  $CH_2$  group; Y = H; Z = H, sulpho, sulphomethyl, or carboxyl; or Y and Z

together = atoms necessary to complete a six-membered isocyclic ring system), e.g. 5-amino-6:8-bisphenylamino-1-naphthalenesulphonic acid, results in the formation of pyrazine dye images.

C. O. C.

**Thioketones and Carbocyanine Dyes.** Kodak Ltd. and R. H. Sprague. B.P. 645,288.

A thioketone and an anhydro *meso*-substituted carbocyanine hydroxide are formed simultaneously by condensing a compound of formula—

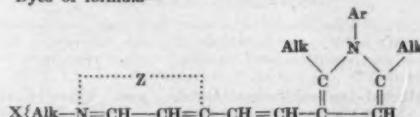


( $R^1$  = carboxyalkyl;  $R^2$  = H or  $CH_3$ ; X = an anion; Z = atoms necessary to complete a benzothiazole, benzelenazole, or *o*-naphthothiazole nucleus) with a dithiocarboxylate of formula  $R^2-CS-SR^4$  ( $R^2$  = hydrocarbon radical;  $R^4$  = alkyl or aralkyl).

C. O. C.

**Pyrrole Dimethincyanine Dyes.** R. H. Sprague, L. G. S. Brooker, and Eastman Kodak Co. U.S.P. 2,515,905.

Dyes of formula—

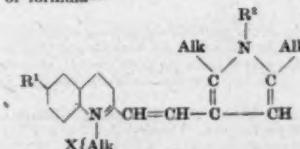


(X = an anion, Z = atoms necessary to complete a quinoline nucleus) are obtained by condensing a suitable pyrrolecarboxylic aldehyde with an appropriate quaternary quinolinium salt.

C. O. C.

**Pyrrole Dimethincyanine Dyes.** E. V. Lare, L. G. S. Brooker, and Eastman Kodak Co. U.S.P. 2,515,912.

Dyes of formula—



( $R^1$  = Alk, Ar, Hal, dialkylamino, or acylamino;  $R^2$  = Alk or Ar; X = an anion) are obtained by condensing suitable pyrrolecarboxylic aldehydes and quaternary quinolinium salts. They are useful as therapeutic agents.

C. O. C.

**Incorporating Selenium in Cadmium Red Pigments.** G. C. Marcot and American Cyanamid Co. U.S.P. 2,515,721.

A method of preparing a calcinable composite is described in which the content of S, Se, Te, and similar non-wettable materials is controlled in amount and properly dispersed, so that on calcination a pigment of optimum quality is obtained.

C. O. C.

**Chemical for Textile Treatment [and Dye Intermediates].** II—*p*-Aminocetylbenzene and Cetyl-*o*-toluidine. Y. Oshima and M. Hayashi. (III, p. 33.)

**Basic Lead Carbonate in Paints.** T. Vignola. (V, p. 39.)

**Dyeing with 3:4-Thioliandithione.** P. D. Caesar and Socony-Vacuum Oil Co. (VIII, p. 43.)

**Pigment Particle Size.** J. H. Martin. (XIV, p. 50.)

**V—PAINTS; ENAMELS; INKS**

**Aluminium Paints.** G. Salomone. *Ind. Vernice*, 2, 125-127 (1948); *Chem. Abs.*, 44, 9159 (10th Oct. 1950).

The loss in brightness and decrease in protective properties which occur in Al paint films on ageing appear to be due to a re-orientation of the Al flakes so that a continuous metallic lamina is no longer present. Treating the Al powder with  $aq.$   $Na_2CrO_4$  (*etc*) before incorporation in the paint increases the period during which the protective structure is maintained, but does not prevent loss of brightness.

C. O. C.

**Basic Lead Carbonate in Paints.** T. Vignola. *Ind. Vernice*, 2, 139-141 (1948); *Chem. Abs.*, 44, 9159 (10th Oct. 1950).

A review of the properties and uses of white lead. It is suggested that the pigment has the structure  $(\text{HO-Pb-CO}_3)_2\text{Pb}$  and that, in paint films, the OH groups are neutralized by the acids present in the medium to form acicular or fibrous crystals of soaps of the type  $(\text{RO-Pb-CO}_3)_2\text{Pb}$ , which reinforce the film, improving its toughness, flexibility, adhesion, and durability. C. O. C.

## PATENT

**Water-set Printing Inks.** A. Vost and J. M. Huber Corp. *U.S.P.* 2,513,122.

Water-set printing inks of increased water tolerance, whose precipitation qualities can be regulated to suit variation in atmospheric humidity, contain as the binder a combination of  $\beta$ -naphthol pitch with a rosin. This binder is dissolved in a water-soluble solvent to provide the vehicle for the pigment. C. O. C.

## VI—FIBRES; YARNS; FABRICS

**Fibres.** C. S. Grove, Jr., J. L. Vodnik, and R. S. Casey. *Ind. Eng. Chem.*, 42, 1983-1986 (Oct. 1950).

A year's progress in the industrial applications of fibre technology is reviewed with emphasis on the impact of the newer synthetic fibres (nylon, Perlon, Orion, Dynel, Vicara, and Terylene). Recent work on fire-retarding, mildew- and rot-proofing, and water-repellency is summarized, and it is noted that increased attention is being given to non-woven fabrics for engineering materials. An annotated bibliography contains 134 references. A. S. F.

**Rôle of Fibres in Archaeology.** J. L. Stoves. *Fibres*, 11, 326-330 (Sept. 1950).

**Some Theoretical Considerations of Dynamic Property Data on Textile Specimens.** W. J. Lyons. *J. Applied Phys.*, 21, 520-522 (1950); *Chem. Abs.*, 44, 8114 (10th Sept. 1950).

(1) Early stretch-vibrometer data on the frequency dependence of the internal friction  $\mu$  of a nylon monocellulose conform to the relation  $\mu = [\mu_0/(1 + \omega^2 T^2)] + \mu_\infty$ , previously deduced by Tobolsky and Eyring (*Chem. Abs.*, 37, 2632 (1943)) from consideration of molecular structure. (2) More sensitive plotting methods than hitherto available confirm by the nylon data that the hyperbolic relationship between resonant frequency  $\omega_0$  and internal friction  $\mu$  holds equally as well as the foregoing equation. (3) With data on 11/4/2 cotton cord the same graphical method establishes the relation  $(\mu + 0.9)\omega_0 = 7.05 \times 10^6 \text{ ergs/c.c.}$  C. O. C.

**Specific Volume of Native Cellulose.** A. J. Stamm. *Text. Research J.*, 20, 631-636 (Sept. 1950).

The specific volume of cellulose calculated from X-ray data represents most closely the true value for this substance. Non-swelling liquids of small molecular size, e.g. benzene, measure the volume of the fibres or the fibre walls but do not penetrate the voids within the walls. On the other hand, helium penetrates voids in the non-crystalline regions and gives values for the specific volume very similar to those calculated from X-ray data. The volume represented by the difference between the specific volume from X-ray data and that measured in water represents a contraction of the water rather than a more complete mechanical penetration of voids. P. C.

**Swelling of Cotton in Water—A Microscopical Study.** A. T. Moore, L. W. Scott, I. V. deGruy, and M. L. Rollins. *Text. Research J.*, 20, 620-630 (Sept. 1950).

A method is described of measuring microscopically the cross-sectional areas of the same fibres before and after wetting, and of comparing their cross-sectional shapes in the wet and dry states. The swelling values of a number of cottons, both normal and immature, and of nylon, viscose rayon, and flax have been determined. Cotton swells 21-34% with an average of 30%. Fortisan about two-thirds as much as cotton, flax and viscose rayon about twice as much as cotton, whilst no swelling of nylon could be detected. Differences in cross-sectional swelling between mature and immature cottons are small and inconsistent, whilst the variations between varieties are

slight. These results imply that, in the selection of cotton for the manufacture of water-resistant fabrics, differences in swelling between varieties play little part. P. C.

**Effect of Moisture on the Frictional Properties of Jute Fibre.** S. B. Bandyopadhyay. *Nature*, 166, 697-698 (21st Oct. 1950).

Samples of jute fibre are conditioned over a wide range of R.H. and their frictional properties measured by fixing a fibre pad on a metal block and allowing it to slide over a similarly mounted pad with an adjustable angle of inclination. Regains of 60-160% are obtained by soaking in water followed by controlled drying. On plotting coefficient of friction  $\mu$  against regain two linear relationships are found, over low and high regain ranges respectively,  $\mu$  being more sensitive to regain over the low range. It is suggested that water molecules on the fibre surface form an inter-pair bond, thus increasing  $\mu$  up to a point, beyond which a multilayer film is formed and the number of molecules contributing to surface bonding falls, thus lowering the slope of the graph. J. W. B.

**Gossypol—Bibliographical Study.** F. C. Aufion. *Bol. Inst. nac. Invest. agron.* (Madrid), 9, 521-555 (1949); *Chem. Abs.*, 44, 7313 (25th Aug. 1950). A review with 45 references. C. O. C.

**Effects of Chemical and Thermal Influences upon Wool.** H. Zahn. *Melliand Textilber.*, 31, 481-486 (July 1950).

An analysis has been made of wool boiled under reflux for periods of from one-half to eight days, and it is shown that a portion of the wool is dissolved with splitting of the cystine and amino acid groups. Paper chromatograms of solutions of "wool gelatin" in *n*-butyl alcohol and *isobutyric acid* have been prepared. "Wool gelatin" is shown to be a mixture of polypeptides, containing approximately one-half of the amount of cystine present in the original wool. Wool fibres and horse hairs were heated dry at 120-210°C., and the contraction was measured during heating, as well as in 5% sodium bimulfite and 20% phenol. The acid-combining capacity, moisture absorption, alkali solubility, and cystine content were also determined. Some samples were hydrolysed and the products examined by partition chromatograms. H. K.

**Non-destructive Mechanical Test for Animal Fibres [Interaction between Concentrated Neutral Salt Solutions and Wool].** H. M. Burte. *J. Applied Phys.*, 21, 494-499 (1950); *Chem. Abs.*, 44, 8114 (10th Sept. 1950).

The slope of the force-extension curve in the Hooke's law region can be measured non-destructively for animal fibres at 3- to 15-min. intervals. Change of this Hookean slope with time is used to follow the course of the heterogeneous reaction between wool and a reagent. Data are given on the interaction between concentrated salt solutions and wool. They indicate that two processes occur—(a) relatively rapid hydration or dehydration of the fibre until equilibrium with the activity of water in the salt solution is attained, (b) relatively slow absorption of salt ions by the fibre. Very concentrated salt solutions dehydrate the fibre so completely that absorption of salt ions is impossible. Swelling experiments and experiments involving other mechanical properties confirm these hypotheses. Interaction between wool and aq. soln. of large organic molecules follows a similar pattern. C. O. C.

**Reaction of Oxidizing Agents with Wool. II—Sorption of Hydrogen Peroxide.** P. Alexander, D. Carter, and C. Earland. *Biochem. J.*, 47, 251-254 (Aug. 1950).

Wool, silk, and nylon, but not cotton, sorb hydrogen peroxide from aqueous solutions. The amount of sorption, which is complete in 1 min., is independent of the pH of the peroxide solution in the pH range 2-5-9, but decreases at both higher and lower pH values. Whilst maximum sorption was not realized in the experiments, a value of 75-100 mg. of  $\text{H}_2\text{O}_2$  per gram of wool is indicated. Acetylated wool sorbs a smaller amount of  $\text{H}_2\text{O}_2$ . It is concluded that  $\text{H}_2\text{O}_2$  is held by both the amino and the peptide groups, the former having the greater affinity, and that the sorption process resembles the formation of complexes with simple organic nitrogen-containing compounds such as urea. F. F. E.

**Composition of Angora Rabbit Wool.** G. Rocchetti. *Ann. sper. agrar. (Rome)*, 4, 273-280 (1950); *Chem. Abs.*, 44, 8113 (10th Sept. 1950).

Commercial Angora rabbit wools of different origins and qualities showed statistically significant differences in the amounts of ethanol-soluble substances, ash, N, and S. Generally the amount of S was higher than in cow hair.

C. O. C.

**Molecular Structure of the Oriented Protein in Muscle.** G. Frank, B. Lemashkin, and V. Kasatochkin. *Doklady Akad. Nauk S.S.R.*, 70, 613-616 (1st Feb. 1950).

X-Ray diffraction patterns of muscle taken at various stages of dehydration indicate that the meridional spots corresponding to a period of 5.1 Å, as observed by Astbury (*Proc. Roy. Soc.*, B 134, 303 (1947)) for living muscle, make their appearance only during dehydration. However, this period was observed on fresh muscle after a considerable dosage of X-rays, and it is possible that it can arise from local structural change, brought about by the intense irradiation. It is concluded that Astbury's contention, that in the contraction of muscle the free energy change is accounted for essentially by the change in internal energy without an appreciable entropy contribution, cannot be considered as proved.

A. E. S.

**Deformation of Proteins of the Keratin-Myosin Group in Solutions of Organic Substances.** A. Pasynsky and V. Blokhina. *Doklady Akad. Nauk S.S.R.*, 73, 535-538 (21st July 1950).

Wool and hair fibres were extended (by 30%) and released while immersed in water, or in solutions of various substances. The work of extension and the area of the hysteresis loop were determined. The experiments were carried out at various temperatures. The solutions used were—0.15 M. acetate buffer (pH 4.1), 5 M. urea, 14% guanidine nitrate, M. urethan, 3% sodium hydrosulphite (pH 4.1), 1% Sulphonol, and 1% Igepon (the last two are surface-active agents). The work of extension was in all cases lower than in pure water, while the relative area of the hysteresis loop was also less, except for hydrosulphite (which causes disulphide breakdown). Similar experiments were carried out on fresh muscle fibre (the whole sartorius muscle from a rat's hind leg). After stretching 30% in physiological soln. (0.9% NaCl) and removing the load, the original length was almost completely regained after 3-4 hr., but on repeating this experiment in 5 M. urea, or in a 0.25% soln. of ATP (adenosinetriphosphoric acid), the work of extension was reduced by 20%, and, on releasing the muscle, 16-17% extension persisted, even after 3-4 hr. If on removing the load the soln. was immediately replaced by 0.9% NaCl, the original length was regained when the treatment soln. was 0.25% ATP, but not when it was 5 M. urea. The hysteresis effect was increased for both ATP and urea, indicating that extensive rupture of intermolecular links had occurred. By studying the way in which the extensibility varied with temp., it was possible to calculate the entropy and internal energy contributions to the load. For hair keratin, extended in water, the entropy contribution was 11.9%; it was 14.0% in 5 M. urea, and 21.2% in 3% sodium hydrosulphite. For wool keratin in 3% sodium hydrosulphite it was 34%. For muscle, in 5 M. urea and in 0.25% ATP, the entropy contribution was considerable—at least 30-40%. It is concluded, in agreement with recent X-ray data (see above), that the purely energetic mechanism of muscle deformation advanced by Astbury (see above) is not correct.

A. E. S.

**Sorption of Non-electrolytes by Proteins.** A. G. Pasynsky and R. S. Chernyak. *Doklady Akad. Nauk S.S.R.*, 73, 771-774 (1st Aug. 1950).

Measurements were made of the degree of adsorption from aq. soln. of urea, guanidine nitrate, and urethan on to various proteins, viz. human serum albumin, human serum globulin, horse serum globulin, gelatin, hair keratin, and wool keratin. The protein and soln. were contained in a collodion bag which was partly immersed in a further quantity of soln., the levels inside and outside being maintained equal. After 4 days at 3-4°C. the change in the concn. of the soln. in the outer vessel was determined, and the apparent adsorption of non-electrolyte calculated. This quantity is a function of the true degree of adsorption of the non-electrolyte and of the degree of adsorption of water. The graph showing the relationship between

apparent adsorption and concn. resembles a full wave of a sine curve, the central portion, which passes across the concn. axis from a region of positive to one of negative apparent adsorption, being practically linear. Such a curve is most readily interpreted on the assumption that there are two kinds of adsorption site, the adsorption of one component of the soln. being confined almost entirely to one kind of site, and the adsorption of the second component to the other kind of site. It is deduced from this curve that the degree of adsorption of water is practically constant for solutions of up to medium concn., and it is therefore possible to construct the true adsorption isotherm for the non-electrolyte. Also, by examining the high-concn. end of the curve it is possible to deduce the max. amount of water adsorbed. The adsorption isotherms found are of the Langmuir type. The max. degree of adsorption of urea corresponds to 1 mol. per 2 amino acid residues for the soluble proteins, and 1 mol. per 8-10 residues for the keratins. The degrees of adsorption of the other non-electrolytes are generally less than those of urea. The values found for the max. degree of adsorption of water are close to the data found in the literature.

A. E. S.

**Kinetics of Degradation of Macromolecular Linear Molecules.** J. Soc. Chem. Ind. Japan, 45, Suppl. binding, 415-417 (1942); *Chem. Abs.*, 44, 7630 (10th Sept. 1950).

**I—Theoretical Equations.** I. Sakurada, S. Okamura, and S. Kawasaki. *Ibid.*, 415.

When  $n = 1$  in the familiar equation  $[\eta] = KP^a$ , the viscosity index of degradation  $m$  is defined and related to the viscosity average mol. wt. by the equation—

$$\frac{1}{m} = \frac{[\eta] \text{ after degradation}}{[\eta] \text{ before degradation}} = \frac{2}{e^a} (s - 1 + \frac{1}{e^a})$$

where  $s$  = average number of bonds broken in one original linear molecule. When  $n = 2$ —

$$\frac{1}{m} = \frac{1}{e^a} (6s - 12 + \frac{12 + 6s}{e^a})$$

When  $n = 0$ ,  $\frac{1}{m} = 1$ . When  $n$  is fractional, as is usually the case,  $m$  can be estimated by interpolating graphically. Experimentally determined average values for  $n$  are—chlorinated polyvinyl chloride in tetrahydrofuran  $\frac{1}{2}$ , polyvinyl chloride in dioxan or tetrahydrofuran  $\frac{1}{2}$ , polyvinyl chloride-acetate in acetone  $\frac{1}{2}$ , polyvinyl acetate in acetone  $\frac{1}{2}$ , polyvinyl alcohol in water  $\frac{1}{2}$ , polymethyl acrylate in acetone  $\frac{1}{2}$ , polymethyl methacrylate in chloroform or acetone  $\frac{1}{2}$ , and polystyrene in benzene 1.

**II—Hydrolysis of Cellulose in Solution.** S. Okamura. *Ibid.*, 416.

The velocity of degradation of cellulose in conc.  $H_2SO_4$  and  $H_3PO_4$  follows the law  $e/t = kN$ , where  $N$  = average degree of polymerization before degradation. In 65%  $H_2SO_4$  at 18°C.,  $k = 4.71 \times 10^{-4}$ ; in  $H_3PO_4$  (sp. gr. 1.640) at 20°C.,  $k = 0.4 \times 10^{-4}$ .

**III—Oxidative Degradation of Polyvinyl Alcohol.** S. Okamura and S. Kawasaki. *Ibid.*, 416-417.

The viscosity of aqueous polyvinyl alcohol is reduced by  $KMnO_4$  or  $K_2Cr_2O_7$  as a second-order reaction, and one active  $O$  atom is consumed for each cleavage of a macromolecule. The data follow the equations in Part I.

**IV—Molecular Weight Distribution.** S. Okamura. *Ibid.*, 417.

The weight fraction  $m_p$  of degradation products smaller than  $P$  (degree of polymerization after degradation) is given by—

$$m_p = 1 - \frac{1}{e^a P} \left[ 1 + aP \left( 1 - \frac{P}{N} \right) \right]$$

where  $a$  = extent of degradation.

**V.** *Ibid.*, 417.

Data on cellulose nitrate and cellulose acetate support the equation in Part IV.

C. O. C.

**Internal Structure of Artificial Fibres. II—Relation between Denier and Mechanical Properties of Viscose Rayon.** T. Kubo and T. Tomonari. *J. Soc. Chem. Ind. Japan*, 44, Suppl. binding, 246 (1941); *Chem. Abs.*, 44, 8113 (10th Sept. 1950).

Orientation of the crystallites in viscose rayon of various deniers spun under identical conditions and elongation

of the fibres decrease with increase in denier, but the tensile strength increases, passing through a maximum. If the fibre is stretched, orientation increases, but the tensile strength decreases unless stretching is done during coagulation and regeneration. C. O. C.

**Load-induced X-ray Line Broadening in Nylon Filaments.** J. Kauffman and W. George. *J. Applied Phys.*, 21, 431-434 (1950); *Chem. Abs.*, 44, 8731 (10th Oct. 1950).

The load-induced microscopic structural changes in highly oriented nylon filaments change the X-ray fibre diffraction. With Type 200 du Pont nylon the change occurs at a strain corresponding to the inflection of the load-strain diagram (14% elongation). The mechanism for smaller plastic strains seems to involve rotation of the crystallized domains into alignment with the fibre axis accompanied by a small overall additional crystallization. For strains larger than the inflectional strain, the mechanism seems to involve progressive reduction in the domain size, and reduction in orientation. Both effects appear to saturation near the rupture strain. C. O. C.

**Orlon.** P. A. Koch. *Textil-Rund.*, 5, 414-417 (Oct. 1950).

The discovery, development, manufacture, properties, and uses of Orlon (polyacrylonitrile fibre) are described in tabular form. F. A.

**Small-angle Interference with Polyurethane Fibres.** H. Zahn and K. Kohler. *Kolloid-Z.*, 118, 115 (Aug. 1950).

Polyurethane filaments give sharp and intense small-angle X-ray interference diagrams. The net plane interval in the direction of the fibre axis is 77 Å. for untreated filaments and up to 122 Å. for filaments which have been heated. F. A.

**Glass Fibre.** P. A. Koch. *Textil-Rund.*, 5, 369-372 (Sept. 1950).

A tabular summary of the preparation, composition, physical and chemical properties, together with the applications and producers of glass fibre. B. K.

#### PATENTS

**Crimped Wool-like Viscose or Viscose-Protein Rayon.** W. D. Nicoll and du Pont. U.S.P. 2,515,834.

The production of crimped viscose rayon of high tensile strength, the crimp of which after mechanical removal, e.g. by stretching or in combing, is completely restored by treatment in an aqueous liquid in absence of tension, is described. U.S.P. 2,515,889.

Similar fibres are produced from viscose solutions containing 5-100% of protein on the weight of cellulose in the viscose. C. O. C.

**Continuously Clarifying and Conditioning the Setting Bath in the Manufacture of Viscose Rayon.** J. A. Calhoun and American Viscose Corp. U.S.P. 2,514,471.

**High-tensile Viscose Rayon of Improved Transverse Properties.** L. G. Ray, Jr., and du Pont. U.S.P. 2,512,908.

Viscose is extruded into a bath which precipitates the cellulose xanthate rapidly without converting it to regenerated cellulose. The unregenerated filaments are highly stretched and then relaxed in presence of a swelling agent. Finally the cellulose is regenerated, and the filaments are purified and dried, preferably without tension. C. O. C.

**Casein Fibre.** R. F. Peterson and U.S. Secretary of Agriculture. U.S.P. 2,512,674.

An alkaline casein solution is extruded into an organic acid, and the precipitated fibres are treated under tension in a hot liquor containing a metal salt and formaldehyde until they are partly hardened and contain 0.2-0.5% combined formaldehyde. They are then immediately immersed in a concentrated buffer solution of pH 6-8. This stabilizes the fibres and further strengthens and hardens them, so that they can be immediately spun into yarn under tension by the "pot" centrifuge method. C. O. C.

**Hardening Casein Fibres.** Courtaulds Ltd., L. E. Hodges, and R. L. Wormall. B.P. 644,958.

A tow of casein is treated with a hardening liquid of sp. gr. > the sp. gr. of the fibres by continuously feeding

it in convolutions on to the surface of the liquid so that the tow floats in the liquid, allowing it to accumulate for sufficient time to produce the desired effect, and then withdrawing the tow from the underside of the mass at a rate equal to the rate of feed; the tow is sprayed with the hardening liquid while it is being fed into the bath to prevent the crystallization of sodium sulphate, which damages the fibres. W. G. C.

**Stretched Collagen Strands.** American Cyanamid Co. B.P. 643,859.

The soft newly spun strand on leaving the spinning bath passes along an endless belt whose linear speed is 20-50% greater than that at which the strand is fed on to the belt. Pretanning, softening, washing, etc. liquids are directed on to the strand while it is on the belt. C. O. C.

**Polyamides having High Resilience.** E. K. Bolton, W. Kirk, Jr., and du Pont. U.S.P. 2,512,606.

These products which have an intrinsic viscosity not > 0.4, which are soluble in an equimolar mixture of  $\text{CHCl}_3$  and  $\text{CH}_3\text{OH}$ , and which are obtained by interaction of equimolecular proportions of a dicarboxylic acid, in which the COOH groups are separated by 4-8  $\text{CH}_2$  groups, and a stereoisomeric mixture of bis-4-aminocyclohexylmethane which is liquid at 25°C., are transparent, clear, and fusible. They are used for fibres, films, and mouldings. C. O. C.

**Nylon Sewing Thread.** Clark Thread Co. B.P. 642,658.

Sewing thread of improved abrasion-resisting quality and high tensile strength is made by applying to each of a number of nylon filaments a twist of  $3.45\sqrt{Y}/(5315/D)$  turns, twisting the yarn together with a finishing twist of  $3.35\sqrt{Y}/(5315/D)$  turns ( $D$  = denier of the yarn;  $Y$  = number of yarns in the thread), and while in a relaxed state treating the thread first in boiling water and then in steam at 245°F. W. G. C.

**Solvents for Nylon.** R. Hill, R. G. A. New, S. M. Todd, and I.C.I. Ltd. B.P. 645,033.

Compounds having 5 atoms linked together to form a single ring, the components of the ring being selected from  $-\text{O}-$ ,  $-\text{CO}-$ ,  $-\text{CHR}-$ , and  $-\text{CR:CR-}$  ( $\text{R} = \text{H}$  or a hydrocarbon of 1 or 2 C), the ring containing at least one O and two CO or alternatively two O and one CO, e.g. maleic, succinic, or phthalic anhydride or ethylene carbonate, are solvents for nylon at moderately elevated temperatures, e.g. 190-200°C. The solutions are clear, pourable, and of relatively high strength. C. O. C.

**Solvents for Aromatic Polyesters.** R. Hill, R. G. A. New, S. M. Todd, and I.C.I. Ltd. B.P. 645,032.

Organic compounds having 4 or 5 atoms linked together to form a single ring structure, the components of the ring being at least one  $-\text{O}-$  and at least one  $-\text{CO}-$  group, the other components being as desired  $-\text{CH}_2-$  and  $-\text{CH:CH}-$  groups, e.g.  $\beta$ -propiolactone, maleic or succinic anhydride, or ethylene carbonate, are good solvents for Terylene and similar polyesters at 150-200°C. C. O. C.

**Films, Filaments, etc. of Crystalline and Oriented Polymers and Copolymers of 2:3-Dichloro-1,3-butadiene.** L. B. Kuhn and Firestone Tire & Rubber Co. U.S.P. 2,514,195.

The production of filaments of high tensile strength, shrink-resistance, and creep-resistance at high temperatures is described. They may be coloured by incorporating pigments or oil-soluble dyes before forming the filament. C. O. C.

**Properties of Solutions of Polyamide Resins.** A. B. Pakshver and T. N. Kokhomskaya. (XIII, p. 47.)

**Dissolution of High Polymers by Mixed Solvents.** A. B. Pakshver, T. N. Kokhomskaya, and R. I. Dolinin. (XIII, p. 47.)

**New Linear Polycondensation Reaction—Transarylation.** V. V. Korshak and G. S. Kolesnikov. (XIII, p. 48.)

## VII—DESIZING; SCOURING; CARBONIZING; BLEACHING

**Studies on Foams. IV—Effect of Electrolyte on the Foam Formation of Congo Red Solution.** M. Nakagaki. *Bull. Chem. Soc. Japan*, 23, 47-49 (July 1950).

With increasing electrolyte concentration, the foam duration of Congo Red solution reached a prominent maximum and then decreased, whilst the amount of foam

was not appreciably affected. The concentration required to achieve maximum foam strength varied with different electrolytes in the same way as the coagulation value. It was considered that the addition of electrolyte decreased the affinity of the polar part of the dye ions for the solvent, resulting in increased surface activity of the solute.

W. K. R.

**Two Experiments on Foam Dispersal.** H. Thiele. *Kolloid-Z.*, 118, 172 (Sept. 1950).

An aqueous solution containing 0.02% of Bromothymol Blue is neutralized to a green colour and aerated to form small foam bubbles, which are yellow. The foam on removal by vigorous passage of air carries the yellow part of the indicator with it, while the residual solution is blue. Foam and residual solution can be recombined to the original green solution. Similarly, an aqueous 0.1% Congo Rubine solution affords a wine-red foam and a violet residual solution. It is concluded that for foam formation there is a definite optimum size of colloid micelles. No such separation takes place with litmus, tropaeolin, or the nitrophenols.

H. H. H.

**Stripping Nylon with Textone (Sodium Chlorite).** W. C. Peurifoy, Jr. *Amer. Dyestuff Rep.*, 39, P 605 (4th Sept. 1950).

Nylon hose is stripped by first treating for 15 min. at 180°F. with 5% dispersing agent, 2% acetic acid (56%), and 6% sulphoxylate, and then rinsing and treating for 15 min. at 180°F. with 5% dispersing agent, 2% acetic acid (56%), and 4% sodium chloride.

F. A.

## PATENTS

**Continuous Hypochlorite Bleaching of Cotton.** L. P. Seyb, J. L. Foster, H. D. Clayton, and Diamond Alkali Co. *U.S.P.* 2,515,532.

A bleach of better whiteness, with less loss of tensile strength and in the same time as or faster than bleaching by peroxide, is obtained by first treating the goods in aqueous hypochlorite containing 0.3-4.5 g. Cl per litre and a wetting and penetrating agent at pH 7.0-9.5, taking them to a holding zone, and then washing with water. They are then treated in 0.5-3.0% aqueous NaOH, passed to a holding zone, scoured, and washed with water. Finally they are given a second hypochlorite treatment under the same conditions as the first, washed with water, anti-chloried, and again washed with water. The goods are continuously passed through the solutions and holding zones, the time of treatment in any solution being not > 1 min. and the time in any one holding zone not > 60 min.

C. O. C.

**Bleaching Cellulosic Materials, particularly Wood Pulp.** G. A. Day, B. G. Hoos, and Brown Co. *U.S.P.* 2,513,787.

Savings in chemicals, stronger pulps without diminished brightness values, and more efficient overall bleaching are obtained by subjecting semi-bleached pulp in one stage of a sequence to the action of a mixture of a hypochlorite and a chlorite. The liquor at the start has pH 9 to inhibit entirely the action of the chlorite; then, when all the hypochlorite has been consumed, the liquor is brought to pH 4 to activate the chlorite.

*U.S.P.* 2,513,787.

The pulp is treated successively, without intervening washing, with hypochlorite and chlorite, NaOH at 140-210°F., and finally at 140°F. and pH 5-7 with chlorine dioxide, the final treatment being carried out at a stock density not > 4%.

C. O. C.

**Bleaching Moist Felted Groundwood Pulp.** R. L. McEwen, F. R. Sheldon, and Buffalo Electro-Chemical Co. Inc. *U.S.P.* 2,514,503.

An alkaline peroxide solution is added to the moist felted high-density pulp so as to reduce the consistency of the latter by < 2-3%. The pulp is then accumulated into a compact mass for storage or shipment. During this period of storage bleaching takes place, and at any time after maximum brightness has been obtained the impregnated pulp can be added directly, without further treatment, to the other paper furnish constituents.

C. O. C.

**Recovery of Grease from Wool Scouring Waste and Abatement of Stream Pollution through the Calcium Hypochlorite Process.** A. A. Cameron. (II, p. 33.)

**Use of Chlorite in the Textile Industry.** H. J. Meybeck. (III, p. 33.)

**Treatment of Wool with Sodium Chlorite.** M. Van Overbeke and G. Mazingue. (X, p. 45).

**Accelerated Washfastness Test.** A.A.T.C.C. Tentative Test No. 3A. (XIV, p. 52.)

## VIII—DYEING

**Permeability of Membranes to Dyes. VI—Dependence of Velocity of Dialysis of Dyes on Temperature. VII—Relation between Dialysis Constant and Particle Size.** T. Nishi. *J. Soc. Chem. Ind. Japan*, 44, Suppl. binding, 233-238 (1941); *Chem. Abstr.*, 44, 8198 (23rd Sept. 1950).

Values of  $k$  for velocity of dialysis of Orange GG, Acid Ponceau E, Double Brilliant Scarlet 3R, and Scarlet 4R were determined at 50, 60, and 70°C. The concentrations of the solutions were determined from the extinction coefficient. In general  $k$  increases linearly with temperature, following the empirical relation of Arrhenius. The dialysis constant varies inversely with mol. wt., but the relation is not precise and can be used only as a measure of relative particle size.

C. O. C.

**Control of the Dyebath by means of Spectrophotometric Measurements.** H. R. Davidson and I. H. Godlove. *Amer. Dyestuff Rep.*, 39, 628-630 (18th Sept. 1950).

Deviations from a standard dyebath composition may be checked by simple spectrophotometric measurements. Full details and examples are given of the method of calculating the corrections required to restandardize a bath containing up to three dyes.

W. K. R.

**Use of Iron and Copper Salts in Dyeing.** E. Justin-Müller. *Teintex*, 15, 401-410 (Sept. 1950).

A critical review of the usages and chemistry of iron and copper salts under the following headings—Iron salts in cotton dyeing, especially for vat dyeing; in wool dyeing, especially for logwood and also in silk weighting. Copper salts as mordants for direct dyes on cotton, Para Brown on cotton, aftertreatment of direct dyes on cotton from an alkaline bath, and for logwood blacks on wool. B. K.

**Effect of Abrasion on the Dyeing Characteristics of Cotton.** K. A. Jenkins and H. F. Keela. *Amer. Dyestuff Rep.*, 39, P 660-P 661 (2nd Oct. 1950).

Knitted cotton material is abraded with sandpaper under standard conditions and dyed with 3 direct dyes, a naphthol dye, and a vat dye, and by a differential technique using red and green dyes. Abraded areas dye much darker, which effect is enhanced if the sample is wetted with water or alkali, while the differential dyeing produces green abraded and red unabraded zones. It is suggested that penetration of dyes is controlled by a thin surface layer relatively impermeable to the larger dye molecules.

J. W. B.

**Studies on Wool Dyeing—Influence of the Cuticle in the Dyeing of the Wool Fibre.** H. E. Millson and L. H. Turl. *Amer. Dyestuff Rep.*, 39, P 647-P 656 (2nd Oct. 1950).

Time-temperature studies of single-fibre wool dyeing under the microscope, using selected dyes, show the dye to be unevenly distributed over the fibre surface, first the distal edges and then perhaps the whole being dyed, while in some cases the edge only is affected. Physical distortion of fibres, produced by tying knots, indicates that the scales become raised from the convex surface of the loop, and dye penetration is more rapid and more complete. A similar effect is found at the most compressed concave portion. It is suggested that the cuticle offers resistance to dye penetration, which takes place through natural or artificial breaks, and is followed by internal diffusion. By suitable adjustment of conditions the above effects can be shown with any type of dye, though dyes known to produce tipped dyeings are most effective. Chlorination attack follows a similar path, and resulting damage to the cuticle leads to much wider areas of penetration during subsequent dyeing.

J. W. B.

**Relations between the Constitution and Suitability for Metachrome Dyeing of *o*-Dihydroxyazo Dyes.** G. Schetty. *Textil-Rund.*, 5, 399-405 (Oct. 1950).

Increase in the number of hydrophilic groups in a dye decreases its affinity for wool. In the metachrome process

a dye must be absorbed in the pH region 5–7. Good metachrome dyes will therefore contain a minimum number of hydrophilic groups. In  $\alpha\alpha'$ -dihydroxyazo dyes one hydroxyl is blocked by chelation with the azo group. Good metachrome dyes are obtained if the second hydroxyl is chelated with a neighbouring sulphonate group, since this causes a mutual decrease in the hydrophilic nature of both these groups. Suitability for metachrome dyeing decreases if the hydroxyl group remains unblocked and if free sulphonate groups are present in the dye. F. A.

**Dyeing of Wool-Polyamide (Nylon, Perlon L) Fibre Mixtures with Acid Dyes.** W. Hees. *Melliand Textilber.*, 31, 496–500 (July 1950).

The excellent properties of mixture yarns of wool and polyamide fibres are briefly described, together with the theoretical considerations involved in the dyeing of polyamide fibres with acid dyes. The difficulties in obtaining level, well penetrated dyeings in solid shades can be overcome by classifying the dyes on the basis of their chemical constitution and absorption characteristics. H. K.

**Dyeing Animalized Cellulose Fibres with Acid Wool Dyes.** P. Eckert and R. Abend. *Kunstseide & Zellwolle*, 27, 385–395, 431–436 (1949); *J. Textile Inst.*, 41, A 417 (Aug. 1950).

Rayon animalized with a mixed resin of phenol, urea, formaldehyde, glycerol, and phthalic acid was dyed with Ponceau 5R and compared with wool. The dyeing characteristics of both fibres are largely dependent on the conditions, and the effects of pH, amount of animalizing resin, dye concentration, and temperature are shown in graphs and tables. Similar series of experiments were carried out with Brilliant Croceine, a diazo dye analogous to Ponceau 5R, but having two instead of three sulphonate groups. In the first few minutes, the rate of absorption of Brilliant Croceine by both fibres is greater than that of Ponceau 5R in the presence of  $H_2SO_4$  and smaller than that of Ponceau 5R in a neutral medium. Total absorption, however, is smaller in both cases for the former dye than for the latter. This difference gradually decreases for animalized staple fibres with increasing initial pH and increasing liquor ratio, whereas it increases for wool with increasing initial pH but decreasing liquor ratio. C. J. W. H.

**Anodizing and Dyeing of Aluminium and its Alloys.** C. C. Hanson. *J. Birmingham Met. Soc.*, 28, 100–127 (1948); *Chem. Abstr.*, 44, 7739 (10th Sept. 1950).

Anodic oxidation of Al and its alloys confers a surface which is corrosion-resistant and has good dye-absorbing and enamelling properties; an oxide film 0.0003 in. thick is satisfactory. Methods of producing these films are described. Films formed in  $H_2SO_4$  are harder and more resistant to abrasion than those formed in  $H_2CrO_4$  or  $CrO_3$ . Dyeing methods with a list of suitable dyes are given. C. O. C.

#### PATENTS

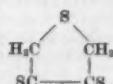
**Improving the Fastness to Water and Washing of Dyeings on Fibres containing Basic Nitrogen Groups.** P. Alexander, J. W. Farmer, and Wolsey Ltd. B.P. 644,213.

The goods are pretreated with aqueous 0.25–1.0% epichlorohydrin at pH 5–9 and 85–98°C., in presence of a small amount of a salt. C. O. C.

**Dyeing Acrylonitrile Polymers with Acetate Rayon Dyes using *m*-Cresol as an Assistant.** J. C. Richards and du Pont. U.S.P. 2,512,969.

Deep and uniform dyeings are obtained by applying dispersed acetate rayon dyes from an aqueous bath containing 1–5% by weight of *m*-cresol. The dyeings have exceptionally good fastness to rubbing and washing. C. O. C.

**Dyeing with 3:4-Thiolandithione.** P. D. Caesar and Socony-Vacuum Oil Co. U.S.P. 2,515,922. 3:4-Thiolandithione—



gives deep purple solutions in alkali. Material dyed in such a solution and then oxidized is dyed a fast red-brown. Wool and silk are directly immersed in the solution and

before drying exposed to an oxidizing atmosphere. Vegetable fibres require mordanting, e.g. with Turkey Red oil or tannic acid-tartar emetic, if a fast dyeing is to be obtained. C. O. C.

**Pad Dyeing with Pigments.** N. S. Cassel, A. T. Clifford, and Interchemical Corp. U.S.P. 2,515,170.

Addition of a hydrophilic glycoside, e.g. a water-soluble non-heat-precipitable cellulose ether or a mannogalactan flour, to a pigmented aqueous solution of a water-soluble heat-precipitable alkyl cellulose and a water-soluble thermosetting resin permits satisfactory control and flocculation of the pigment on drying to be obtained with much less of the heat-precipitable alkyl cellulose present than would otherwise be necessary. The dyeing is rendered wash-fast enough by drying the padded fabric on steam-heated drying cans or through a low-temperature oven to enable it to be printed with a vat or azo dye and given the necessary acid- or steam-aging before curing the resin. C. O. C.

**Package Dyeing.** E. A. Stienen. B.P. 644,658.

An improved and economical method of uniformly dyeing packages consists in producing a predetermined pressure in a dye liquor in a closed system independently of pressure apparatus therein, and then causing the liquor to circulate at this pressure by a low-pressure means, e.g. use of a propeller which causes a pressure differential of 5 lb./sq. in. The pressure in the system may be first produced by use of simple water-main pressure, and then steam injected to cause the water pressure to rise when needed. C. O. C.

**Daylight-fluorescent Textiles.** J. L. Switzer. B.P. 644,201.

Fluorescent fabrics of clean and pure colour under either visible light or invisible ultra-violet radiation are obtained by dyeing cellulose ester rayon fabrics, whose fibres are translucent, in a dilute solution of a daylight-fluorescent dye and/or a dye fluorescent under ultra-violet radiation in a strong aqueous solution of a solvent for the dye, which solvent is also a swelling agent for the rayon. The temperature of the dyebath, the concentration of dye in it, and the time of dyeing are such that a maximum quantity varying from 0.25 g. of dye projecting yellow-green light to 0.015 g. of dye projecting red light is absorbed per c.c. of fibre. The temperature of the bath and the time of dyeing are also such that the filaments almost reach a state of incipient coalescence. C. O. C.

**Prediction of Dye Formulae and the Bluing of White Papers.** H. J. Selling. (XIV, p. 51.)

**Relationship between the Amount of Dye used and Colour Sensation obtained and its Possible Application in Textile Dyeing.** H. Cikurel. (XIV, p. 51.)

#### IX—PRINTING

**Studies in Textile Printing. I—Evaluation Methods and Terminology.** F. Fordemwalt and C. R. Witschonke. *Amer. Dyestuff Rep.*, 39, 607–609 (4th Sept. 1950).

When evaluating the results of printing processes it is necessary to measure the amount of dye applied in the printing operation, the amount of dye fixed in the fibre, and the colour value. From these measurements it is proposed to calculate three factors known as the Percent Fixation, the Colour Efficiency, and the Printing Efficiency. It is hoped that the proposed terminology will assist in work designed to elucidate the mechanism of printing. F. A.

**Pigment Printing.** R. Haller. *Textil-Rund.*, 5, 405–412 (Oct. 1950).

Methods which have been used for the pigment coloration of textiles are reviewed. Microscopic examination shows that the pigments fall into two groups—The older type, which include Turkey Red, Manganese Bistre, and Paranntraniline Red, give a continuous film on the fibre surface, and the pigment particles become visible only when this layer is disrupted by swelling the fibre. These pigments are usually formed *in situ* from components with a high degree of dispersion and are retained on the fibre surface by van der Waals forces. The latest Aridye and Orama dyes, however, consist of coarser particles of

pigment, which are deposited within a discontinuous resin layer on the fibre surface.

F. A.

## PATENTS

**Colour-stable Water-in-Lacquer Printing Emulsions containing Copper Phthalocyanine.** Interchemical Corp. *B.P.* 643,724.

Printing emulsions containing copper phthalocyanine dispersed in a water-in-lacquer emulsion whose outer phase is a heat-convertible synthetic resin soluble in aromatic hydrocarbons and insoluble in saturated aliphatic hydrocarbons, dissolved in a volatile solvent free from aromatic hydrocarbons and from unsaturated aliphatic hydrocarbons and consisting mostly of pine terpene, do not tend to lose tinting strength on standing, as do such emulsions containing unsaturated hydrocarbons. This is because copper phthalocyanine is very slightly soluble in and recrystallizes from many unsaturated hydrocarbon solvents which must be used.

C. O. C.

**Printing Pastes of Water-in-Lacquer Type.** Interchemical Corp. *B.P.* 644,390.

Printing pastes of maximum stability and minimum solids content comprise an aqueous dye solution dispersed in a solution of several film-forming substances in a volatile water-immiscible organic solvent. One of the film-forming substances must be a natural or synthetic rubber, and the film-forming substances must not be > 5% of the total composition. Such emulsions do not flush in printing, and yield the fine lines of a photogravure etching even on materials such as cotton flannel.

C. O. C.

**Preventing Staining with Indophenol or Azomethin Dye Images.** H. C. Harsh, J. E. Bates, and General Aniline & Film Corp. *U.S.P.* 2,515,121.

Stains on indophenol or azomethin dye images produced in multilayer colour film by colour development are obviated if the film is rinsed after fixing and washing but before drying with an aqueous open-chain mono-, di-, or tri-basic acid.

*U.S.P.* 2,515,122.

Use as the fixing agent of an aqueous solution of an alkali sulphite and ammonium or sodium thiosulphate prevents staining.

C. O. C.

**Diazotype Prints of Two or More Colours.** Hall Hardinge Ltd. and W. P. Leuch. *B.P.* 644,493.

Two or more diazo compounds are used, at least one of which cannot form a dye with the azo coupling components present in the light-sensitive layer (in the case of layers developed by ammonia gas) or in the developer (in the case of moist or semi-dry development) but can couple with its own decomposition product produced by exposure to light. At least one other diazo compound is used, which can couple with the azo coupling component contained in the light-sensitive layer or developer. The quantity of each diazo compound is such that sufficient difference in printing-out speeds results between at least two of the diazo compounds used, so that at least two colours are produced side by side and not superimposed.

C. O. C.

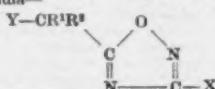
**Screen Printing.** J. S. Wheelwright and John Wheelwright & Co. Ltd. *B.P.* 644,474.

The printing bed has a slot through which the part of the fabric upon which a pattern unit has been printed is passed on the conclusion of such printing operation. This enables the next successive pattern unit to be printed upon that part of the fabric immediately following that just printed, as a continuation of the pattern, without any risk of smearing or blurring the wet pattern unit immediately preceding it.

C. O. C.

**Colour Photography.** P. L. J. R. Merckx and Gevaert Photo-Products N.V. *B.P.* 645,043.

A reducible silver salt image is developed with a primary aromatic amino developing agent in presence of a compound of formula—



( $\text{R}^1$  = a substituent capable of being split off during development, or it may represent a second such oxadiazole group linked directly or through S, S-S, or subst. or unsubst.  $\text{CH}_2$ ;  $\text{R}^2 = \text{H}$  or  $\text{R}^1$ ; X = an organic radical; Y = an electronegative radical).

C. O. C.

**Application of Transfers or Prints to Pottery, etc.**Edwards & Jones Ltd. and J. J. Reed. *B.P.* 644,762.

**Cyanoacetamides as Azo Coupling Components in Diazo-type Layers.** General Aniline & Film Corp., W. H. von Glahn, and L. N. Stanley. (IV, p. 37.)

**Photographs in Blue-Black Tones.** General Aniline & Film Corp., F. Dersch, and N. Heimbach. (IV, p. 37.)

**Studies in Textile Printing. II—Analytical Methods for Determining the Vat Dye Contents of Textile Prints.** C. R. Witschonke and F. Fordemwalt. (XIV, p. 51.)

## X—SIZING AND FINISHING

**Preparation of Water- and Alkali-soluble Cotton Yarns.** J. D. Reid, G. C. Daul, and R. M. Reinhardt. *Text. Research J.*, 20, 657-659 (Sept. 1950).

The authors draw attention to the distinction between alkali-soluble metal carboxymethyl cellulose fibres and the water- and alkali-soluble modified cotton yarns. The former are prepared by extruding a solution of sodium carboxymethyl cellulose into a coagulating bath containing a metal salt the metal of which reacts with the modified cellulose to form a water-insoluble salt. The modified cotton yarns are produced by carboxymethylating cotton yarn so that it becomes water- and alkali-soluble without altering greatly the intrinsic yarn structure and fibrous characteristics. A typical method of preparation of the latter type is described.

P. C.

**Effect of Weather on Bleached and Unbleached Cotton Duck.** J. D. Dean, W. N. Berard, and R. K. Worner. *Text. Research J.*, 20, 643-649 (Sept. 1950).

On exposure to light and weather for periods up to 12 months, the average strength losses for grey cloth (duck) were higher and more erratic than those for a-scoured bleached duck, owing to the presence of impurities in the former fabric which rendered it susceptible to non-uniform and seasonal variations in microbiological attack. Conversely, the average fluidity increase was less for the grey than for the bleached fabric. pH values decreased gradually from about 8.0 to 6.0. The grey fabric had an initially high copper number, but after a brief exposure the average copper numbers of both fabrics were equal, and afterwards that of the bleached cloth was consistently higher.

P. C.

**Some Felting Properties of Wools of Different Geographical Origins.** A. M. Sookne, H. Bogaty, and M. Harris. *Text. Research J.*, 20, 637-642 (Sept. 1950).

The felting properties of different wools under laundering conditions have been studied using wool principally in top form. The mechanical and frictional characteristics of the fibres were also measured. Only small differences in felting behaviour were found among the 11 wools examined, such differences being correlated with staple length, the longer fibres promoting felting. No correlation between fibre diameter and felting was observed. Laundering tests on socks made from 4 pure wools of different geographical origin confirmed the findings of the top felting tests.

P. C.

**Enzymatic Digestion of Protein Fibres.** M. T. Goldsmith. *Text. Research J.*, 20, 613-616 (Sept. 1950).

By culturing an actinomycete of the genus *Streptomyces*, a cell-free proteolytic enzyme preparation was obtained having the power of decomposing wool and man-made protein fibres such as peanut, soya-bean, and casein fibres. Wool and soya-bean are only slightly digested, whereas casein bristle and peanut protein fibre show marked susceptibility. The stability of protein fibres to enzymatic digestion imparted by the manufacturing process is not of the same order as that found in the protein of wool, soya-bean being the exception to this rule.

P. C.

**Cross-linkage Formation in Keratin.** C. Fearnley and J. B. Speakman. *Nature*, 166, 743-744 (28th Oct. 1950).

Lincoln wool fibres are treated with 2.5% solutions of 1:2:3:4-diepoxybutane (I) and 1:2:5:6-diepoxyhexane (II) in a phosphate buffer for 24 hr. at 50°C. Cross-linkage formation is estimated by determining the extent of

supercontraction on boiling for 30 min. in 5% sodium metabisulphite soln. At pH 4.44–7.87 reagent (I) effectively prevents supercontraction, thus showing cross-linkage formation; reagent (II) has little or no effect, which may be due to furan-ring formation after the primary reaction with the carboxyl groups of wool. J. W. B.

**Treatment of Wool with Sodium Chlorite.** M. Van Overbeke and G. Mazingue. *Bull. Inst. Text. France*, (18), 11–24 (1950); *J. Textile Inst.*, 41, A 470 (Sept. 1950).

An investigation of the value of sodium chlorite in processing wool is described. The wool did not appreciably deteriorate if the sodium chlorite treatment took place under the following conditions—pH 5–9, temp. not exceeding 50°C., max. concn. of technical sodium chlorite on the weight of wool 1%, min. liquor ratio 60 : 1, and max. duration of treatment 2 hr. The process was of little use as a bleach for wool. Wool, however, received a certain degree of unshrinkability by treatment with 0.5% concentration of sodium chlorite for 2 hr. at pH 7–9 and 40°C., the liquor ratio being 50 : 1. C. J. W. H.

**Anomalous Results of Mothproofing Tests.** R. W. Moncrieff. *Nature*, 166, 659 (14th Oct. 1950).

Explanations are given for the findings of Lamb (cf. *J.S.D.C.*, 66, 499 (Sept. 1950))—(1) Application of mothproofing agents from acid solution at 60°C. secured normal behaviour because the fabric had acquired a good proof which gradually diminished in efficiency on removal of agent by repeated washing. (2) Application along with a dye from a neutral bath at 100°C., followed by acidification immediately prior to removal of fabric, gives an unsatisfactory proofing because the agents, which are substantive as sulphonate acids, will attach themselves irregularly owing to insufficient time for thorough penetration. Mothproofing agents of the type described by Lamb are, in effect, colourless acid dyes. (3) Washing with a commercial soap powder may lead to a redistribution of the agent, still present on the wool as the free sulphonate acid, and so improve the quality of the proof. H. H. H.

**Hexamethylene Diether of 1-Hydroxymethyl-pyridinium Chloride Cross-linking Agent—Application in Rayon Finishing.** J. Harms. Zellwolle, Kunsteide, Seide, 47, 730–732 (1942); *Chem. Abs.*, 44, 8118 (10th Sept. 1950).

The chloride reacts with air-dry or freshly spun cellulose rayon, apparently to establish cross-linkages. The rayon is steeped in a 1–10% soln. containing sodium acetate to neutralize free HCl, squeezed or hydroextracted, dried, and baked at 130°C. The strength of the wet fibre is increased 30–40%, solubility in 10% aq. NaOH decreased from 30 to 1.5%, and swelling in water reduced from 90 to 50%. The finish is water-repellent and resists boiling water. Effects of a similar agent prepared from decamethylene are compared graphically with those of the hexamethylene anil. C. O. C.

**Characteristics of Textiles treated [with Synthetic Resins] to produce Crease-resistance.** E. Cerbaro. *Boll. cotoneira*, 37, 176–178 (1942); *Chem. Abs.*, 44, 8118 (10th Sept. 1950).

Cloth impregnated with 12.2% of urea-formaldehyde resin was cured, washed with 0.5% aq. soap soln. at 15°C., and then dried at 100–105°C. until constant in weight. When cured at room temperature, the loss in weight on washing, i.e. removal of unpolymerized resin, fell from 5.5% after one day to a constant 1.5% after 4 days. Curing at 80–140°C. for 5–60 min. resulted in the best results (loss in weight of 0.7–0.8%) from 1 hr. at 100°C. or 40 min. at 140°C. Treatment of spun viscose rayon fabric increased dry tensile strength *T* of warp and weft by 32.1 and 19.1% and wet strength *W* by 48.3 and 36.2% respectively. Corresponding values after 10 washes were *T* 17.6 and 24.6, *W* 45.9 and 47.6 respectively. C. O. C.

#### PATENTS

**Mercerizing Single-ply Cotton Yarns.** W. F. Luther and Dixie Mercerizing Co. *U.S.P.* 2,512,951.

The yarn is wound on a perforated tube, the tension being gradually increased as winding proceeds. The wound package is then treated with mercerizing lye. C. O. C.

**Preshrinking Fabrics.** F. R. Redman. *B.P.* 643,714.

Modification of *B.P.* 571,821 (cf. *J.S.D.C.*, 62, 87 (1946)). The fabric is repeatedly subjected over its entire area to compressive rumpling, in the direction of its length only, in the presence of moisture and heat, so as to reduce the tension on it, the fabric being kept free of tension during and between the rumplings. C. O. C.

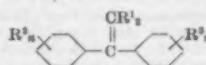
**Moiré Effects.** A. Holterhoff. *U.S.P.* 2,513,646.

Moiré effects of most varied types are obtained if the fabric is moistened within confined areas while being kept under tension: e.g. the fabrics are moistened with a pattern and dried while under tension, and then doubled and hot-pressed to produce the final moiré effects. The resulting moiré patterns are superior to those produced by the "scratch" method, particularly in the case of acetate rayon fabrics. C. O. C.

**Rendering Wool-containing Textiles resistant to Shrinking and to the Attacks of Carpet Beetle Grubs.** C. A. Amick and American Cyanamid Co.

*U.S.P.* 2,515,107.

The material is impregnated with a mixture of (1) an alkylated methylolmelamine partly or completely soluble in water and (2) a compound of formula—



( $\text{R}^1 = \text{Br or Cl}; \text{R}^2 = \text{H, Br, Cl, Alk, or hydroxy-alkyl}; n = 1 \text{ or } 2$ ), the proportion of (1) : (2) being by weight 1 : 0.05–0.5. After drying and baking, the resulting shrink-resistant and mothproof finish is fast to washing. C. O. C.

**Rendering Wool-containing Textiles resistant to Shrinking and to Attack by Moths and Other Insects.** R. H. Kienle, C. A. Amick, and American Cyanamid Co. *U.S.P.* 2,514,132.

The materials are impregnated with an aqueous composition containing (1) an alkylated methylolmelamine which is at least partly, and preferably completely, soluble in water, e.g. a water-soluble methylated polymethylolmelamine, and (2) a melamine fluorosilicate and/or melamine fluoroborate. There should be 0.05–0.5 part by weight of (2) for each part of (1). After drying and baking, the resultant shrink-resistant and mothproof finish is fast to washing. C. O. C.

**Reducing the Felting Properties of Wool.** F. R. Haigh, H. Ehrhardt, E. C. Kourner, and Botany Worsted Mills. *U.S.P.* 2,516,055.

Wool is rendered non-felting by partly halogenating it and then applying a resin, e.g. an aqueous dispersion of polymerized olefins, neither treatment being of itself sufficient to render the wool non-felting. The treated material has a soft handle. Dyeing may be carried out in the normal manner before or after the chlorination or after the resin treatment, the finished product having the same hue and fastness properties as would be obtained on untreated material. C. O. C.

**Increasing the Resistance of Wool to Decomposition by Heat.** C. R. Barnes and Albany Felt Co. *U.S.P.* 2,515,181.

The heat resistance of wool is improved by treatment in an aqueous solution containing complex metallo-thiocyanate ions, the symmetrical zinc thiocyanate complex giving the most effective results. The metal thiocyanate forms a cross-linkage at the amine groups of the wool and leaves the disulphide linkage intact. C. O. C.

**Papermakers' Felt.** Orr Felt & Blanket Co. *B.P.* 644,088.

Impregnation with a silicone resin extends the service life of papermakers' felt, particularly as regards abrasion, and does not unduly extend its wetting time. C. O. C.

**Treating Polyurethane or Polycarbamide Fibres with Formaldehyde.** S. B. McFarlane, G. E. Moos, and Celanese Corp. of America. *U.S.P.* 2,514,550.

Treating the fibres in an aqueous solution of formaldehyde or a formaldehyde-yielding substance in presence of a catalyst of pH either < 3.5 or > 10 at 50–150°C., then thoroughly washing with distilled water, and drying

results in increased m.p., regain, and lustre, and, in the case of fibres which have been previously cold-drawn, imparts increased elasticity. The increase in regain imparts a desirable handle and a soft, silky appearance. C. O. C.

**Non-woven Pile Fabrics.** H. Ewing. *B.P. 644,002.*

A base fabric is given a sticky coating of a thermoplastic polymer and two plasticizers of different volatilities. The fibres forming the pile are then applied, and the assembly is heated to evaporate the more volatile plasticizer and so render the coating non-sticky and flexible. C. O. C.

**Milling.** E. B. Bates. (I, p. 32.)

**Swelling of Cotton in Water—A Microscopical Study.** A. T. Moore, L. W. Scott, I. V. deGruy, and M. L. Rollins. (VI, p. 39.)

**New Wool-destroying Fungus—*Ctenomyces* Species.** S. R. S. Gupta, S. S. Nigam, and R. N. Tandan. (XIV, p. 52.)

**Fishy Smell in Goods finished with Urea- or Melamine-Formaldehyde Resins—Detection of Trimethylamine.** W. Kraus. (XIV, p. 52.)

**Testing the Resistance of Mothproofing Treatments to Dry Cleaning and Washing.** Schweizerischer Verband für die Materialprüfungen der Technik, Kommission 25, Richtlinien-Entwurf A 3011. (XIV, p. 52.)

## XI—PAPER AND OTHER CELLULOSIC PRODUCTS

**Acid-soluble Derivatives of Cellulose.** D. S. Breslow. *J. Amer. Chem. Soc.*, **72**, 4244-4246 (Sept. 1950).

Ethyl cellulose- $\beta$ -pyridylcarbamate, cellulose acetate- $\beta$ -pyridylcarbamate, and hydroxyethyl cellulose- $p$ -dimethylaminophenylcarbamate are all soluble in dilute acids. They are prepared by treating the appropriate cellulose derivative with nicotinyl azide or  $p$ -dimethylaminophenyl isocyanate. C. O. C.

**Hydroxyethyl Cellulose and its Uses.** W. E. Gloor, B. H. Mahlman, and R. D. Ulrich. *Ind. Eng. Chem.*, **42**, 2150-2153 (Oct. 1950).

The properties and uses of various hydroxyethyl celluloses are summarized. The variety having a degree of substitution of 0.35-0.4 is a white fibrous solid, soluble in alkaline 40% urea solutions, which may be cast into films from solutions in 7% caustic soda. The literature on end uses of alkali-soluble hydroxyethyl celluloses is surveyed under the headings—filament forms, sheet forms, textile finishes and sizes, paper coating, pigment binders, and miscellaneous uses (e.g. in mineral flotation and leather coating). A. S. F.

**Splitting of Lignin by Metallic Sodium in Liquid Ammonia—IV.** N. N. Shorygina and T. Ya. Kefeli. *J. Gen. Chem. (U.S.S.R.)*, **20**, 1199-1208 (July 1950).

The decomposition of lignin by means of metallic sodium in liquid ammonia results in the formation of a number of monomeric substances. From these a phenolic substance was isolated in 13% yield on the lignin taken. It was shown to be 1-*p*-hydroxy-*m*-methoxyphenyl-2-propanol. A. E. S.

**Pure Titanium Dioxide in Papermaking.** P. Lusseyran. *Papeterie*, **72**, 305, 307, 309, 311 (1950); *Chem. Abs.*, **44**, 8107 (10th Sept. 1950).

A discussion of the use of  $TiO_2$  in improving the quality of various types of paper. C. O. C.

### PATENTS

**Coating Compositions particularly suitable for Paper.** R. Tutt, Jr., J. R. Hubbard, and Peter Cooper Corpsa. *U.S.P. 2,513,121.*

Clay is dispersed in water containing a deflocculating agent. A solution of an alkali or alkaline salt is then slowly added until the pH is 8.5-11.5. Any auxiliary pigment to be used is then added in either a dry or a dispersed state, and the whole made into a smooth uniform mixture. Animal glue solution is then added. The product has a

high solid content with a desirable fluidity, and while having an alkaline pH does not hydrolyse or degrade the glue. It gives an unusually fine coating on paper or other materials. C. O. C.

**Coated Paperboard.** H. C. Fisher, B. E. Sooy, and Gardner Board & Carton Co. *U.S.P. 2,515,340.*

A plasticizing, film-forming, water-borne substance chemically inert to the fibres is applied to freshly made paper as it passes through a roller nip. Then, while the surface is still damp, a coating of a mineral coating material and a starch binder in an aqueous vehicle is imprinted on to the paper. The coating is rendered resistant to wet rubbing by adding to it a thermosetting resin and a catalyst therefor to produce a reaction in which the starch is insolubilized. Neither the resin nor the catalyst is applied to the paper before the plasticizer, but at least one of them must be applied before the mineral coating. Colouring matter can be applied to the paper simultaneously with any of the above. C. O. C.

**Coating Paper.** Time Inc. *B.P. 643,747.*

A viscous coating containing about 70% by weight of solids is uniformly distributed over moving surfaces whilst the respective vapour pressures of the coating material and of the surrounding atmosphere (humidified by introducing steam) are maintained so as to prevent evaporation of moisture from the coating material, and the coating material is transferred to the web. The moving surfaces are constituted by a series of distributing rollers, alternately heat-conductive and non-conductive, at least one of the rollers being cooled to approximately the dew-point of the surrounding atmosphere to prevent substantial evaporation of moisture from the coating material. S. V. S.

**Coating Paper, etc. to produce a Uniform Surface.** Sponjéné Papierne N.P. *B.P. 644,789.*

Excess of an aqueous coating medium of viscosity not < 500 centipoises and of solids content > 33% is applied to a rotating surface having small pits or cavities of uniform size regularly distributed over it. The excess is doctoried off, and the contents of the cavities are transferred to a non-absorbent elastic surface; the small heaps so deposited then flow together to form a coherent layer, with which the paper is brought into contact under rolling pressure. C. O. C.

**Tub-sizing Paper using Citric or Tartaric Acid.** R. N. Woodward and Eastman Kodak Co. *U.S.P. 2,514,689.*

When citric or tartaric acid is employed as the catalyst in sizes of the glue-urea-formaldehyde resin type, the sized paper is free from frilling, in contrast to papers prepared using inorganic catalysts. C. O. C.

**Embossed Wallpaper.** E. A. F. van Leer. *B.P. 644,514.*

In manufacturing embossed wallpaper by the pressure printing system the paper is led between a pick-up roll rotating in a coating bath and an adjustable smooth roll determining the thickness of the coating, after which the coated web is passed beneath an embossing roll. The pick-up roll and the embossing roll may be arranged vertically on either side of the smooth roll, or an impression cylinder operating in conjunction with the embossing roll may be utilized, the line of centres of the pick-up roll and the smooth roll being substantially perpendicular to the tangential path of the paper from the smooth roll to the impression cylinder. S. V. S.

**Water-detecting Paper.** M. S. Kantrowitz and E. J. Gossell. *U.S.P. 2,515,232.*

A paper which becomes strongly coloured when wetted, e.g. by writing on it with an aqueous invisible ink, is obtained by use of a coated composition consisting of an organic dye which is practically colourless in thin layers but which develops strong coloration when wetted, a drying oil, a lower alkyl ether or lower alkanone of cellulose, and an organic solvent for the drying oil and the cellulose derivative. *C.I. 671* is the most suitable dye, but *C.I. 518*, 520, and 715 may also be used. C. O. C.

**Carbon Papers.** C. A. Hampshire and Columbia Ribbon & Carbon Manufacturing Co. Ltd. *B.P. 644,142.*

Defective ageing and maturing of carbon paper while in roll or package may be eliminated by backing it with an oil-repellent coating. C. O. C.

**Emulsions of Stearic Acid.** Nopco Chemical Co. (III, p. 34.)

**Preparation of Water- and Alkali-soluble Cotton Yarns.** J. D. Reid, G. C. Daul, and R. M. Reinhardt. (X, p. 44.)

**Dissolution of High Polymers by Mixed Solvents.** A. B. Pakshver, T. N. Kokhomskaya, and R. I. Dolinin. (XIII, below.)

**Prediction of Dye Formulas and the Bleaching of White Papers.** H. J. Selling. (XIV, p. 51.)

## XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

**Electron-microscopic Investigation of Collagen by the Replica Method.** A. L. Zaides and S. L. Pupko. *Doklady Akad. Nauk S.S.R.*, **73**, 379-380 (11th July 1950).

Dry samples of collagen were examined under the electron microscope, using the polystyrene-quartz replica technique. For wet samples a methyl methacrylate-quartz technique was evolved—the methyl methacrylate was applied at a low degree of polymerization, and was allowed to polymerize further while in contact with the specimen. A table is given showing the observed periodicity of the transverse striations on the fibrils and the width of the fibrils for collagen which has been subjected to various tanning and other treatments. Electron micrographs, showing the effects of some of these treatments, accompany the text. They indicate that the positions of the striations of neighbouring fibrils are related, the dark or light bands on one fibril lying precisely in line with the corresponding bands of the next.

A. E. S.

**Electron-microscopic Investigation of the Action of Alkali and Pancreatin on Collagen.** A. L. Zaides and S. L. Pupko. *Doklady Akad. Nauk S.S.R.*, **73**, 991-992 (11th Aug. 1950).

Sections of collagen, which had been treated for various times with milk of lime or with pancreatin, were dispersed by the action of sound waves, mounted on collodion film, and examined under the electron microscope. Electron micrographs accompany the text.

A. E. S.

## PATENTS

**Degreasing Sheep and Goat Skins or the like.** S. Friestedt. *B.P.* 645,040.

Pickled skins which have been limed and puered are treated in a bath of ammonia of such strength that after the pickle acid has been neutralized the bath is still alkaline. The excess of ammonia forms soap with that portion of the fat which has been hydrolysed to free fatty acids and glycerol during liming.

C. O. C.

**Tanning Agents.** I. Bolgar. *B.P.* 644,451.

Improved tanning sulphonic acids are obtained by condensing an aromatic sulphonic acid with a urea-(or thiourea)-ammonia-aldehyde condensate containing an active hydroxymethyl group and an amino group.

C. O. C.

**Resorcinol-Aldehyde Tanning Agents.** H. C. Beachell and du Pont. *U.S.P.* 2,512,708.

The low-mol.wt. copolymers of resorcinol and a ketaldione (aldehyde or ketone) together with 50-150% by weight of boric acid or an alkali borate form a tanning agent which gives improved penetration of skins and decreased grain drawing.

C. O. C.

**Molecular Structure of the Oriented Protein in Muscle.** G. Frank, B. Lemzhiukhin, and V. Kasatochkin. (VI, p. 40.)

**Deformation of Proteins of the Keratin-Myosin Group in Solutions of Organic Substances.** A. Pasynsky and V. Blokhina. (VI, p. 40.)

## XIII—RUBBER; RESINS; PLASTICS

**Properties of Solutions of Polyamide Resins.** A. B. Pakshver and T. N. Kokhomskaya. *J. Appl. Chem. (U.S.S.R.)*, **23**, 837-846 (Aug. 1950).

An examination was made of solutions of a polycondensation product of caprolactam. The specific viscosity,

the specific volume change accompanying dissolution, and the apparent density of the polymer, calculated on the basis of an additive law of mixture, were determined for solutions of the resin in 92-5%  $H_2SO_4$ , 80% formic acid, 90% phenol, and 80% phenol. The amounts of resin dissolved on immersing, in the form of fibre or fine powder, in various solvents at various temperatures for definite periods of time were also determined. The solvents used included conc. solutions of organic acids, alcohols, alcohol-water mixtures, and solutions of calcium chloride in methanol or in a mixture of methanol and water. The results are presented in the form of tables and graphs and are discussed in their relation to the nature of the intermolecular forces in the polymer and the solvating properties of the solvents. The specific viscosity varies considerably from one solvent to another, but the change in overall volume which occurs on dissolution is very small and is not always negative. This is contrasted with the case of cellulose acetate solutions (cf. *J. A.D.C.*, **66**, 663 (Dec. 1950)), where there is a marked specific volume contraction, which is closely linked with the specific viscosity. The difference in behaviour is related to the shorter chain length and more open array of polar groups found in the polyamide. In contrast to the case of cellulose acetate, the interaction between neighbouring polar groups of the same chain is small; the molecule is therefore flexible and readily takes up a coiled form which is not strongly solvated. The addition of the monomer or acetone to a soln. of the polyamide in formic acid leads to complete solvation of the CO-NH groups, and so to a reduction in viscosity. The "power" of the various classes of solvent, as indicated by the viscosity and solubility figures, falls off in the following order—formic acid > phenols > mineral acids > fatty acids > alcohols > water.

A. E. S.

**Dissolution of High Polymers by Mixed Solvents.**

A. B. Pakshver, T. N. Kokhomskaya, and R. I. Dolinin. *J. Appl. Chem. (U.S.S.R.)*, **23**, 900-997 (Sept. 1950).

An examination was made of solutions of a secondary cellulose acetate and of a polyamide resin (polycaprolactam) in binary mixtures, consisting of a solvent for the polymer in admixture either with another solvent, or with a non-solvent which, when used by itself, causes swelling only. The mixtures used for cellulose acetate were acetone-acetic acid and acetone-ethanol. The mixtures used for the polyamide were phenol-formic acid, phenol-water, and formic acid-water. As in the previous investigations of the authors in this field (see above), determinations were made of the specific viscosity of the soln. and of the apparent density of the polymer in soln. Whereas the specific viscosity of solutions of polymer in solvent-solvent mixtures varies with composition in an approximately linear manner, in solvent-non-solvent mixtures the specific viscosity falls sharply as the concn. of non-solvent increases and, in the case of cellulose acetate solutions, passes through a minimum value and rises again. The reduction in viscosity attained is very considerable for the polyamide solutions (30-50% reduction compared with solutions in pure solvent); it is accompanied by a large initial rise in the apparent density of the polymer in soln., followed by a fall to the initial value as the concn. of solvent (phenol) is reduced further. The results are explained by considering the interaction of the solvent molecules with the highly associated molecules of hydroxyl non-solvent, leading to a reduction in the degree of association of the non-solvent and so to the liberation of unassociated molecules which are able to complete the solvation of the polar groups in the polymer molecule.

A. E. S.

**Polymer containing Free Aldehyde Groups.** E. F. Izard. *Ind. Eng. Chem.*, **42**, 2108-2110 (Oct. 1950).

Copolymerization of vinyl acetate and allylidene diacetate yields a product which on alkaline alcoholysis gives a water-soluble product containing free hydroxyl and aldehyde groups. This may be applied to paper or fabrics and insolubilized by heating or treatment with a mild acid catalyst. Potential uses of the polymer are to increase the wet strength of paper and to anchor pigments to paper or fabrics to produce solid colorations or printed effects.

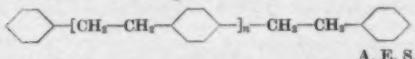
A. S. F.

**Crystallization of Polyurethan.** E. Jenckel and E. Klein. *Kolloid-Z.*, 118, 86-93 (Aug. 1950).

Polyurethan crystallizes from a melt in the form of spherulites. By staining and examination with polarized light the spherulites are shown to contain a small central nucleus from which radiate bands of molecules oriented in the same direction as tangents to the spherulites. F. A.

**New Linear Polycondensation Reaction—Transarylation.** V. V. Korshak and G. S. Kolesnikov. *Doklady Akad. Nauk S.S.R.*, 70, 625-627 (1st Feb. 1950).

When 1,2-diphenylethane is heated with anhyd. aluminium chloride in a stream of carbon dioxide, benzene is eliminated, and a linear polycondensate, of mol. wt. ~4000, of the following structure is formed—



PATENTS

A. E. S.

**Stabilizers for Rubberlike Copolymers of Butadiene and Acrylonitrile.** H. E. Albert and Firestone Tire & Rubber Co. *U.S.P.* 2,514,220.

Alkylated naphthols are used as non-discolouring stabilizers. C. O. C.

**Alkylated Naphthols as Anti-flexcracking Agents for Natural Rubber.** H. E. Albert and Firestone Tire & Rubber Co. *U.S.P.* 2,514,218.

**Stannous Phenylcatechoxides as Stabilizers for Rubberlike Conjugated Diene-Vinyl Compound Copolymers.** H. E. Albert and Firestone Tire & Rubber Co. *U.S.P.* 2,514,221.

**Extenders for Chloroprene Polymers and Interpolymers.** D. H. Coffey, O. B. Edgar, T. J. Meyrick, J. T. Watts, and I.C.I. Ltd. *B.P.* 644,829.

Polyisocyanate-modified polyesters, e.g. hexamethylene diisocyanate-modified polyethylene sebacate, are excellent extenders for incorporation with chloroprene polymers or interpolymers before curing. They do not readily volatilize or migrate from the composition. C. O. C.

**Linear Polyesters.** Wingfoot Corp. *B.P.* 642,423. Polyesters of high mol. wt. are prepared by heating a dicarboxylic acid with excess of a glycol (2-15% over equimol. proportions) and then adding the acid chloride of a dibasic acid to complete the condensation. W. G. C.

**Rapid-curing Melamine Resins.** British Industrial Plastics Ltd. and H. P. Wohnsiedler. *B.P.* 643,750.

A melamine-formaldehyde condensate is spray-dried, compounded with accelerators, and, if desired, pigments, plasticizers, and lubricants but no fillers, and is then hot-milled and granulated to a partly precured moulding powder. The granules, which cure rapidly under heat and pressure, may be tableted and are suitable for moulding into buttons, etc. resistant to heat, water, and laundering. E. C.

**Kinetics of Degradation of Macromolecular Linear Molecules. I—Theoretical Equations.** I. Sakurada, S. Okamura, and S. Kawasaki. (VI, p. 40.)

**Polymides having High Resilience.** E. K. Bolton, W. Kirk, Jr., and du Pont. (VI, p. 41.)

**Films, Filaments, etc. of Crystalline and Oriented Polymers and Copolymers of 2:3-Dichloro-1:3-butadiene.** L. B. Kuhn and Firestone Tire & Rubber Co. (VI, p. 41.)

#### XIV—ANALYSIS; TESTING; APPARATUS

**Ion Exchange Resins in Chromatography.** C. W. Davison. *Research*, 3, 447-452 (Oct. 1950).

Review with 17 references. C. O. C.

**Determination of Iron in Commercial Sulphuric Acid.** R. Doht. *Chem. Listy*, 40, 266, 267 (1946); *Chem. Abs.*, 44, 9300 (25th Oct. 1950).

To remove  $\text{SO}_4^{2-}$  from commercial  $\text{H}_2\text{SO}_4$ , which would interfere with determination of Fe, 100 ml. of  $\text{H}_2\text{SO}_4$  is

diluted to 500 ml. with water, 0.5-1.0 ml. of  $\text{H}_2\text{O}_2$  is added, and the excess removed with  $\text{KMnO}_4$ . The  $\text{Fe}^{3+}$  is reduced to  $\text{Fe}^{2+}$  and titrated by the Zimmermann-Reinhardt method. C. O. C.

**Physicochemical Characterization of Wetting Agents.** E. Bartholomé and K. Schäfer. *Melliand Textilber.*, 31, 487-491 (July 1950).

Surface tension measurements alone are not completely satisfactory for expressing speed of wetting of wetting agents, but if it is assumed that the change of surface tension with time is conditioned by the diffusion of wetting agent molecules through the solution to the surface, then measurement of change of surface tension with time after production of a new wetting surface provides a method for determining speed of wetting. Change of surface tension with time is represented graphically for a number of wetting agents. A formula is derived from which it is concluded that there is a relationship between molecular structure and speed of wetting. H. K.

**Drop-spreading Test for Investigating Wetting Phenomena.** W. Neudert. *Kolloid-Z.*, 118, 113-114 (Aug. 1950).

The relation between the volume and the shape of a stationary drop on a horizontal surface is determined. The apparatus consists of a microburette which allows a drop to be built up in increments and a scale of concentric circles for measuring the radius of the drop. The shape of the graph relating the volume and radius of the drop depends on the wettability of the surface and the wetting power of the liquid. F. A.

**New Approach to the Evaluation of Wool Oils for Resistance to Oxidation in Storage.** A.A.T.C.C., Mid-west Section. *Amer. Dyestuff Rep.*, 39, P 633-P 638 (18th Sept. 1950).

In an interlaboratory investigation, patterns oiled with eleven wool oils, varying from excellent to poor in resistance to natural ageing, were subjected to—(a) natural ageing for 1 year; (b) accelerated ageing at 120°F. for 3 weeks, (c) accelerated ageing at 190°F. for 16 hr., and (d) Fadometer exposure for 20 hr. The patterns were then examined for colour and for the effect of scouring and subsequent dyeing. Excellent correlation was obtained between natural ageing and accelerated ageing at 120°F., whilst the correlation between Fadometer exposure and natural ageing and between individual Fadometers was poor. The 190°F. ageing test gave unsatisfactory results with oils of fatty ester type, but otherwise could be used as a screening test. It is recommended that the 120°F. accelerated ageing test should replace the Fadometer test for evaluating the oxidizing characteristics of wool oils. W. K. R.

**Potentiometric Determination of Chlorite.** R. Wiener. *Z. Elektrochem.*, 52, 234-237 (1948); *Water Pollution Abs.*, 23, 33 (Feb. 1950).

Difficulties in determining chlorite are discussed and a simple method of potentiometric titration with  $\text{FeSO}_4$  is described. To avoid losses by volatilization of gaseous  $\text{ClO}_2$  from acid solution, the determination is done in weakly alkaline solution, sodium acetate being added to neutralize mineral acids formed by hydrolysis and added with the  $\text{FeSO}_4$  during titration. The end-point is clear, and the approach to it can be followed from the potentiometric readings even without drawing a curve. Ammonium acetate reacts with chlorite and so is unsuitable for buffering. The method satisfactorily separates chlorite from chlorate, chloride, and carbonate.  $\text{H}_2\text{O}_2$  interferes and is best removed by boiling with glass powder. C. O. C.

**Purity Tests for Thiocyanates.** E. Boye. *Pharm. Ztg.*, 86, 460 (1950); *Chem. Abs.*, 44, 10,263 (10th Nov. 1950).

$\text{KCN}$  and  $\text{NH}_4\text{CN}$  down to 0.005% can be detected in thiocyanates by dissolving a 1-g. sample in water, adding 2 ml. picric acid solution and 1 ml. aq.  $\text{NaOH}$  (concn. not given), boiling, and allowing to stand. A reddish coloration indicates  $\text{CN}^-$ . C. O. C.

**Spot Test for Copper with o-Nitrosophenol.** M. Polster. *Chem. Listy*, 43, 288, 289 (1949); *Chem. Abs.*, 44, 9300 (25th Oct. 1950).

The Baudisch-Rotschild reaction of Cu with o-nitroso-phenol (obtained as a light green solution in petroleum

ether by dissolving a few crystals of *o*-nitrophenol in 1 ml. 96% acetic acid, heating, diluting with 3 ml. distilled water, cooling, adding 5 ml. petroleum ether and some Zn powder) is used as a spot test for detecting Cu in textiles, water, and sterilizing preparations. The neutral or acidic solution to be tested is dropped on filter paper, and a drop of the reagent added. In presence of Cu a pink spot appears. Ni, Cu, Bi, Hg, and Fe also give a positive test, but the colour is less intense.

C. O. C.

**Colorimetric Determination of Small Quantities of *m*-Dinitrobenzene in some Aromatic Mononitro Compounds.** M. Jurecek. *Chem. Listy*, **43**, 204, 205 (1949); *Chem. Abs.*, **44**, 9304 (25th Oct. 1950).

Aromatic nitro compounds containing 2 or 3 nitro groups in *meta* position give with NaOH in acetone solution intense red, violet, or blue colorations. Compounds of the *ortho* or *para* series do not react. The groups—OH, O-CO-CH<sub>3</sub>, NH<sub>2</sub>, NHR, NR<sup>2</sup>, NH-CO-CH<sub>3</sub> interfere; O-CH<sub>3</sub>, COOH, and COOR do not. 2:4-Dinitromesitylene, trinitromesitylene, and triminto-1:3-dimethyl-5-butylbenzene give no colour reaction. The method was worked out for determining *m*-dinitrobenzene in excess of *o*-, *m*-, and *p*-chloronitrobenzene and nitrobenzene. Attempts to use the method in presence of nitrophenols were unsuccessful.

C. O. C.

**Indirect Colorimetry—Determination of the Apparent Dissociation Constants of Mononitroquinolin, 2:6-Dinitroquinolin, and 3-Alizarinsulphonic Acid.** L. Sacconi. *J. Phys. & Colloid Chem.*, **54**, 829-841 (1950); *Chem. Abs.*, **44**, 8745 (10th Oct. 1950).

Indirect colorimetry makes possible determination of  $pK$  values when it is impossible to isolate both the optical  $H$ I and  $I^-$  forms of an indicator. In the double incomplete colour change method, instead of two standard solutions, each containing one of the two coloured forms, two fictitious standard solutions, included within the range of the colour change, are used. In the simple incomplete colour change method, when only one optical form can be isolated, a fictitious standard within the colour change range is used. A wedge colorimeter of the Bjerrum-Kolthoff type is employed. The upper field of the movable cell and the lower field of the joined wedge cells are brought to optical contiguity by a prism arrangement. For mononitroquinolin,  $pK_1$  is  $7.63 \pm 0.02$  by the simple incomplete colour change method and  $7.65 \pm 0.02$  by the double incomplete colour change method, and  $pK_2$  is  $10.06 \pm 0.02$ ; for 2:6-dinitroquinolin,  $pK_1$  is  $4.42 \pm 0.02$  in one series and  $4.43 \pm 0.03$  in another series of experiments, and  $pK_2$  is  $9.14 \pm 0.03$ ; for 3-alizarinsulphonic acid  $pK_1$  is  $5.54 \pm 0.01$  and  $pK_2$  is  $11.01 \pm 0.04$ . For both mononitroquinolin and 2:6-dinitroquinolin a yellow form does not exist, and the green-yellow to orange colour change is simple.

C. O. C.

**Capillary Analysis of Dyes—I and II.** F. Yokoyama. *J. Pharm. Soc. Japan*, **63**, 492-532 and 595-611 (1943); *Chem. Abs.*, **44**, 8055 (10th Sept. 1950).

Capillary analysis allows determination of dyes present in very small samples of drugs (5-10 mg.). A method based on that of Itoh (*Chem. Abs.*, **34**, 2137 (1940)) is described, and examples are given of its use.

C. O. C.

**Determination of Acetates, Carbonates, Halides, and Sulphates in Certified Coal Tar Products. [Determination of Sodium Acetate in FD&C Blue No. 1.]** J. Schifferli and A. T. Schramm. *J. Assoc. Off. Agric. Chem.*, **33**, 385-386 (1950); *Chem. Abs.*, **44**, 9154 (10th Oct. 1950).

The modified Sclar and Clark method (cf. *J.S.D.C.*, **66**, 253 (April 1950)) has given satisfactory results in a collaborative study.

C. O. C.

**Determination of Ether Extract in Coal-tar Colours.** S. S. Forrest. *J. Assoc. Off. Agric. Chem.*, **33**, 397-398 (1950); *Chem. Abs.*, **44**, 9117 (10th Oct. 1950).

Use of isopropyl ether instead of ethyl ether as the extracting solvent by various methods has given considerably varied results in a collaborative study.

C. O. C.

**Determination of Boiling Range of Amines derived from Coal-tar Colours. [Evaluation of the Boiling Range of Xylidine in FD&C Red No. 32.]** L. S. Harrow. *J. Assoc. Off. Agric. Chem.*, **33**, 413-416 (1950); *Chem. Abs.*, **44**, 9155 (10th Oct. 1950).

The intermediate is recovered from the dye by reduction with Zn and HCl, and the boiling range determined in a semimicro fractionating apparatus.

C. O. C.

**Report on [Determination of] Volatile Amine Intermediates in Coal-tar Colours—Xylidine.** A. B. Caemmerer. *J. Assoc. Off. Agric. Chem.*, **33**, 384-385 (1950); *Chem. Abs.*, **44**, 8063 (25th Sept. 1950).

Report on a collaborative study of a method for determining any xylidine present (*ibid.*, **31**, 81 (1948)). All the results were within the expected experimental error.

C. O. C.

**Determination of Non-volatile Unsulphonated Amine Intermediates.** L. S. Harrow. *J. Assoc. Off. Agric. Chem.*, **33**, 390-396 (1950); *Chem. Abs.*, **44**, 9154 (10th Oct. 1950).

A titration method (cf. *J.S.D.C.*, **66**, 253 (April 1950)) is extended to include determination of 2-nitro-4-methoxyaniline in Ext. D&C Orange No. 1 and 2-chloro-4-nitroaniline in D&C Red No. 36. Spectrophotometric methods are detailed for determination of aminonitrobenzene in D&C Red No. 17, aminoxazoline in D&C Red No. 18, and 2:4-dinitroaniline in D&C Orange No. 17.

C. O. C.

**Determination of *p*-Phenylenediamine by Diazotization.** M. D. Shorina. *Zavodskaya Lab.*, **13**, 1180-1181 (1947); *Chem. Zentr. (Russian Zone Ed.)*, **1**, 495 (1948); *Chem. Abs.*, **44**, 7716 (10th Sept. 1950).

Sodium nitrite diazotizes only one NH group of *p*-phenylenediamine, an amide being probably formed. Under certain conditions this reaction is quantitative. An aqueous solution of a 4-g. sample is acidified with 4 c.c. conc. HCl and diluted to 500 c.c. To 50 c.c. of this solution are then added 5 c.c. conc. HCl and 35 c.c. conc. acetic acid. The solution is then cooled to 5°C. and titrated with 0.1 N- $\text{NaNO}_2$ . When starch-iodide paper remains coloured for 5 min. the reaction is considered complete.

C. O. C.

**Determination of *p*-Phenylenediamine and 2:5-Diaminotoluene in Hair Dyes and Rinses.** S. H. Newburger and J. H. Jones. *J. Assoc. Off. Agric. Chem.*, **33**, 374-379 (1950); *Chem. Abs.*, **44**, 9119 (10th Oct. 1950).

Details are given of a method for analysing mixtures of *p*-phenylenediamine and 2:5-diaminotoluene in hair dyes. It is based upon the spectrophotometric properties of alcoholic solutions of diacetyl derivatives of these diamines, optical density measurements being made at 250 and 270 m $\mu$ .

C. O. C.

**Determination of Unsulphonated Phenolic Intermediates in Coal-tar Colours. [Determination of  $\beta$ -Naphthol in D&C Red No. 35 and Orange No. 17.]** H. Holtzman and H. Graham. *J. Assoc. Off. Agric. Chem.*, **33**, 387-390 (1950); *Chem. Abs.*, **44**, 9154 (10th Oct. 1950).

A method involving continuous extraction with isopropyl ether, separation with dil. NaOH, and coupling with diazotized *p*-nitroaniline has given satisfactory results in a collaborative study.

C. O. C.

**Identification of Organic Dyes by X-Ray Powder Diffraction.** G. V. Sushich. ORR 314/50\* (PB 99,530).

The technique of the method is described, together with treatment of specimens; polymorphism is discussed, and there is a section on methods of comparison by standard samples. Limitations of the method are outlined. Examples of identification are described and illustrated with reference to sensitivity of technique, detection of polymorphism, and solution of special problems. Other methods of identification by means of dye tests, chemical investigation, and heated-specimen microscopy are also dealt with.

C. O. C.

\* Photocopies available from T.I.D.U., Board of Trade, London, Theobalds Road, London W.C.1, or (under PB No.) from Office of Technical Services, Department of Commerce, Washington D.C.—see *J.S.D.C.*, **66**, 53 (Jan. 1950).

**Spectrophotometric Testing of Coal-tar Colours.**

J. H. Jones. *J. Assoc. Off. Agric. Chem.*, 33, 401-405 (1950); *Chem. Abs.*, 44, 9155 (10th Oct. 1950).

Collaborative study of the photometric determination of the pure dye content of four samples of certifiable dyes is described. The results for three of them were satisfactory, but the fourth sample apparently changed in composition between the time it was prepared and the time several of the collaborators analysed it; hence, the results with this sample could not be evaluated. Results obtained with each of the several spectrophotometers used show good agreement as to wavelength of maximum absorption of solutions of the dyes, but significant differences in the specific extinction coefficients. C. O. C.

**Spectrophotometric Analysis of Coal-tar Colours—**

**D&C Orange No. 17.** R. N. Sclar. *J. Assoc. Off. Agric. Chem.*, 33, 418-424 (1950); *Chem. Abs.*, 44, 9118 (10th Oct. 1950).

Chloroform solutions of pure D&C Orange No. 17 follow Beer's law. The absorption peak is at  $479 \pm 2 \text{ m}\mu$ . The extinction per mg./litre at this wavelength is  $0.0767 \pm 0.0004$ . The extinction ratio  $E_{450\text{m}\mu}/E_{479\text{m}\mu}$  is  $0.96 \pm 0.01$ . The data obtained with chloroform solutions of the dye were applied to the determination of the pure dye content of commercial samples. C. O. C.

**Determination of Subsidiary Dyes in D&C Colours,**

**D&C Red No. 6 or 7.** L. Koch. *J. Assoc. Off. Agric. Chem.*, 33, 405-407 (1950); *Chem. Abs.*, 44, 9155 (10th Oct. 1950).

A previous method (cf. J.S.D.C., 66, 165 (Feb. 1950)) has been slightly modified. Collaborative study has shown the modified method to be superior. C. O. C.

**Determination of Subsidiary Dyes in FD&C Colours.**

M. Dolinsky. *J. Assoc. Off. Agric. Chem.*, 33, 407-409 (1950); *Chem. Abs.*, 44, 9117 (10th Oct. 1950).

A method for determining the amount of D&C Orange No. 4 present in D&C Orange No. 1 (cf. J.S.D.C., 66, 253 (April 1950)) has given satisfactory results in a collaborative study. C. O. C.

**Determination of the Pigment in Mascaras, Eyebrow**

**Pencils, and Eye Shadow.** P. W. Jewel. *J. Assoc. Off. Agric. Chem.*, 33, 359 (1950); *Chem. Abs.*, 44, 9119 (10th Oct. 1950).

A technique for extracting the base from the pigment is described. C. O. C.

**Microchemical Differentiation and Recognition of Mineral Colours.** S. Augusti. *Ind. Vernice* (Milan), 3, 134-143 (1949); *Chem. Abs.*, 44, 9693 (25th Oct. 1950).

A systematic scheme of microanalysis (under the microscope or by spot tests) for identifying pigments generally found in ancient, medieval, and modern paintings. C. O. C.

**Microchemical Investigation of the Anions in Mineral Pigments taken from Paintings.** S. Augusti. *Ind. Vernice* (Milan), 3, 226-229 (1949); *Chem. Abs.*, 44, 9694 (25th Oct. 1950).

Detailed spot tests for identifying chromates, carbonates, sulphates, sulphides, antimonates, acetates, phosphates, and silicates are given. C. O. C.

**Microchemical Investigation of Cations for Identifying Mineral Colours in Paintings.** S. Augusti. *Ind. Vernice* (Milan), 3, 178-188 (1949); *Chem. Abs.*, 44, 9695 (25th Oct. 1950).

Detailed tests for identifying Ca, Zn, Ba, Pb, As, Sb, Fe, Hg, Cr, Cd, Al, Cu, Co, and Mn, and their sensitivities are described. C. O. C.

**Pigment Particle Size.** J. H. Martin. *Paint.* 20, 405-408.

A review of present methods of measurement and the influence of the particle size of pigments on paint systems. There are 14 references. C. O. C.

**Spectrophotometric Methods of Establishing Empirical Formulae of Coloured Complexes in Solution.** A. E. Harvey and D. L. Manning. *J. Amer. Chem. Soc.*, 72, 4488-4493 (Oct. 1950).

A new slope ratio method and a modified molar ratio

method are described and compared with other spectrophotometric methods for determining formulae of coloured complexes. C. O. C.

**Determination of Water in Shellac.** A. Wright. *Paint.* 20, 349-352, 356 (Oct. 1950).

Karl Fischer reagent is used for a rapid and very accurate method for quantitatively determining the water in natural and bleached shellac. The preparation of the reagent is discussed, and also several techniques which may be used to carry out the determination. The results obtained by this and several other methods are compared. C. O. C.

**Analysis of Driers.** J. Gavarret. *Congr. tech. intern. Ind. Peintures Inds. Assoc.*, 1, 203-205 (1947); *Chem. Abs.*, 44, 9697 (25th Oct. 1950).

An outline of a method for the quantitative analysis of non-aqueous solutions of metallo-organic salts. Non-volatile content is determined by distilling off the solvent under reduced pressure, adding a low-boiling solvent to the residue, and redistilling. This last operation is repeated until the weight of the residue is constant. The metal is extracted from the liquid drier by diluting with benzene or trichloroethylene, adding a suitable mineral acid, and refluxing for 3-4 hr. Co, Cu, Mn, and Zn are extracted by  $\text{H}_2\text{SO}_4$  or  $\text{HNO}_3$ , Pb by  $\text{HNO}_3$ , and Mg by a mixture of fuming  $\text{HNO}_3$ , HCl, and  $\text{KClO}_3$ . The acid layer, containing the metals, is separated, and analysed by volumetric methods. A micro method is given for determining Cu and Hg in very low concentrations in aqueous solutions. C. O. C.

**Practical Application of Microchemical Methods in the Examination of Artists' Paints.** H. Malissa and H. Shitzy. *Mikrochemie ver. Mikrochim. Acta*, 35, 302-305 (1950); *Chem. Abs.*, 44, 7718 (10th Sept. 1950).

Microtests are described which enabled pigments used in 1740-42 to be identified, and which showed them to be quite different from those used in restoring the same painting in 1929. C. O. C.

**Rapid Separation of Amino Acids by Two-dimensional Ascending Paper Chromatography.** R. A. Boissonnas. *Helv. Chim. Acta*, 33, 1966-1971 (Oct. 1950).

A paper-chromatographic method for separating mixtures of 18 amino acids in less than 13 hr. is described, based on the use of solvents of low viscosity. Leucine, isoleucine, valine, methionine, phenylalanine, tyrosine, and tryptophan are separated from each other by the systems of solvents—*tert*-butyl alcohol-methyl ethyl ketone-water (4 : 4 : 2) and *tert*-butyl alcohol-methanol-water (4 : 5 : 1). The other acids (aspartic and glutamic acids, alanine, arginine, asparagine, cystine, glycine, histidine, lysine, proline, serine, and threonine) are separated by the systems phenol-water (7 : 3) and *n*-propanol-water (7 : 3). Removal of solvent is effected below  $60^\circ\text{C}$ . H. H. H.

**Point Identification of Amino Acids, Polypeptides, and Sugars separated by Paper Chromatography.** R. A. Boissonnas. *Helv. Chim. Acta*, 33, 1972-1974 (Oct. 1950).

The above substances, when separated on paper, are localized by point development at distances of  $\sim 7 \text{ mm}$ . from each other. This method is very sensitive and leaves 99% of the amino acid, etc. intact. H. H. H.

**Colorimetric Determination of Amino Acids separated by Paper Chromatography.** R. A. Boissonnas. *Helv. Chim. Acta*, 33, 1975-1982 (Oct. 1950).

Moore and Stein's reagent, ninhydrin-hydridantin (cf. *J. Biol. Chem.*, 176, 367 (1948)), has been used for colorimetric determination of amino acids separated by paper chromatography. The ammonium ions in the paper are eliminated previously by vaporization from a solution of potassium methoxide. The method enables 0.02-0.5 micromoles of amino acids to be estimated. H. H. H.

**Quantitative Analysis of Powders by Infra-red Spectrophotometry.** G. Pirlot. *Bull. Soc. chim. Belg.*, 59, 327-351 (June-July 1950).

The compensated extinction coefficient method for the analysis of powders when the Beer law is not obeyed, and

when there is some overlapping between the absorptions of the different components of the mixture, is discussed mathematically, and then applied to mixtures of alkaloids and of DL-leucine, DL-isoleucine, and DL-norleucine. There are 13 curves, 7 tables, and 29 references. H. H. H.

**Identification of Fibres.** J. M. Preston. *J. Textile Inst.*, 41, p. 679-P 691 (Sept. 1950).

An account is given of useful techniques for fibre identification. Preliminary tests include handle, wet and dry manual tensile tests, ease of wetting, estimation of staple length, and odour. Contraction temperatures and m.p. of different fibres are summarized as an addition to the usual combustion tests. Data necessary for density determination by flotation are included, while a micro-swelling method and factors influencing swelling properties are discussed. Consideration of X-ray diffraction patterns and optical anisotropy is of value, though practical application is usually not feasible. Staining tests and microscopic examination are briefly summarized. J. W. B.

**Quantitative Analysis of Textiles containing Asbestos, Glass Fibres, and Organic Fibres.** G. Satiow. *Glastech. Ber.*, 22, 121-127 (1949); *Chem. Abs.*, 44, 10,277 (10th Nov. 1950).

A quantitative method which is simpler and quicker than microscopic analysis is based on determining the ignition loss of serpentine asbestos (chrysotile), which is taken to average 14.5%. By dehydrating at 900°C. for 1 hr., differentiation between asbestos and glass fibres is possible. If, however, they are mixed with organic fibres, a first calcination at 500°C. must be done for 1 hr. to destroy the organic fibres and a second 1-hr. ignition at 900°C. to dehydrate the asbestos. Mineral wool is equivalent to glass fibres in this test. Canadian, Rhodesian, Italian, and Russian specimens of asbestos behave alike on calcination. Amphibole asbestos is assumed not to be used in textiles, because its lower thermal stability permits dehydration at 450-500°C. C. O. C.

**Measurement of Fabric Wrinkling.** H. H. Hebel and H. J. Kolb. *Text. Research J.*, 20, 650-653 (Sept. 1950).

An instrument for determining the wrinkle-resistance and wrinkle-recovery of fabrics is described. A standard size of fabric is wrinkled or creased by stuffing in a random manner into a tube and held under a known pressure for a given time. The fabric is removed, and spread out on a movable platform which passes steadily underneath a counterbalanced probe. The latter traces electronically a diagram of the hills and valleys in the surface of the creased fabric. From the diagram produced the mean wrinkle height  $\bar{h}_0$  is derived. Wrinkle-resistance index is denoted by  $10/\bar{h}_0$  (mm.<sup>-1</sup>) and wrinkle-recovery index by  $100(\bar{h}_0 - \bar{h}_1)/\bar{h}_0$ , where  $\bar{h}_0$  and  $\bar{h}_1$  are the mean wrinkle heights immediately after wrinkling and after 4 days' recovery. Typical results are quoted. P. C.

**Foam Measurement.** J. G. Sinsheimer. *Soap Sanit. Chemicals*, 26, (8), 38-41, 157 (1950); *Chem. Abs.*, 44, 9701 (23th Oct. 1950).

A review of the theory and measurement of foam formation. The sudsing test given in Federal Specification P-S-536 was modified by using a mechanical shaker and an improved test cylinder, and recording the initial and final volumes of the foam for 15 min. following the shaking. The dynamic foam meter method of Hatiangdi *et al.* (*Chem. Abs.*, 43, 6435 (1949)) was modified by regulating the pressure of the gas used to produce the foam and calibrating the apparatus. A factor was derived for each bubbler and used for correlating data. C. O. C.

**Printing Soil Cloth.** C. Z. Draves and O. L. Sherburne. *Amer. Dyestuff Rep.*, 39, P 771-P 772 (13th Nov. 1950).

A printing paste containing soiling ingredients is prepared from Stock Paste A, comprising a mixture of lampblack, hydrogenated fat, and a heavy water-white mineral oil, and Thickening Paste B, comprising solvents, e.g. water or butanol, thickeners, e.g. sodium alginate gum, and emulsifiers, e.g. oleic acid and morpholine. A and B

are mixed and applied under standardized printing conditions to standard cotton material, which is then cured and used for the detergency tests. The method is extended to other cloths, including wool, silk, and nylon, for which curing temperatures are given. Cured soil cloths may be stored without deterioration, and excellent reproducibility is claimed. J. W. B.

**Prediction of Dye Formulae and the Bleaching of White Papers.** H. J. Selling. *Proc. Tech. Sect. British Paper & Board Makers' Assocn.*, 31, 271-312 (June 1950).

A discussion of colour and its measurement with the spectrophotometer and the visual and photoelectric colorimeters is followed by a consideration of the prediction of dye formulae based on the following series of operations—(1) Determine the specifications of the given sample with a simple photoelectric colorimeter: e.g.  $X = 14,500$ ,  $Y = 14,100$ ,  $Z = 15,500$  (time needed about 5 min.). (2) Select the graphs for the measured  $X$ ,  $Y$ , and  $Z$  values. (3) Arrange the graphs (drawn on transparent paper) in such a way that the squares cover each other. Draw a line through points of corresponding yellow concentration in the  $X-Y$  combination and do the same in the  $X-Z$  combination ( $\sim 5$  min.). (4) Read the recipe determined by the point of intersection of the two lines. Whiteness and its measurement are then discussed, including measurement of the whiteness of blued papers and application of whiteness formulae. S. V. S.

**Objective Measurement of Colour and Colour Change.** B. Buchmann-Olsen. *Tids. Textiltek.*, 8, 94-99 (1950); *Chem. Abs.*, 44, 8171 (25th Sept. 1950).

A description of an objective trichometer, having Se barrier-layer photocells and based on simple filtering, follows a review of existing methods of colour measurement. This instrument enables speedy and accurate determination of C.I.E. tristimulus values and has been successfully used in the textile industry. C. O. C.

**Automatic Colorimeter for Cotton.** D. Nickerson. *J. Optical Soc. Amer.*, 40, 446-449 (1950); *Chem. Abs.*, 44, 8114 (10th Sept. 1950).

An instrument based on satisfactory application of the Hunter Color and Color-Difference Meter to measuring raw cotton. It is fully automatic and self-standardizing, and shows graphically values for reflectance and yellowness on a two-dimensional chart. Although this model is limited to measuring within the range of cotton colour, its design is adaptable to other limited ranges of colour, for which it provides an automatic, self-standardizing, small-difference colorimeter. C. O. C.

**Relationship between the Amount of Dye used and Colour Sensation obtained and its Possible Application in Textile Dyeing.** H. Cikurel. *Amer. Dyestuff Rep.*, 39, P 736-P 738 (30th Oct. 1950).

Samples of wool fabric are dyed with different quantities of dye, and it is shown that a logarithmic relation exists between colour sensation and amount of dye, i.e. a range of strengths having a common difference do not appear to be evenly reduced in shade, whereas those with a common ratio do. This is an illustration of the Weber-Fechner law of the relation between stimulus and sensation. J. W. B.

**Studies in Textile Printing. II—Analytical Methods for Determining the Vat Dye Contents of Textile Prints.** C. R. Witschonke and F. Fordenwalt. *Amer. Dyestuff Rep.*, 39, 621-624, 646 (18th Sept. 1950).

The vat dye content of either unaged or developed prints may be determined by extracting the oxidized dye with boiling anhydrous pyridine, and measuring the concentration of the extract spectrophotometrically at 60°C. by comparison with a standard transmission curve for the dye involved. To obtain complete extraction, finished patterns are wetted with water prior to extraction, and in some cases, e.g. Blue 2B type, the addition of a small quantity of a swelling agent such as calcium thiocyanate is advantageous. Unaged prints are first neutralized by treatment with dil. acid. The vat dye content of unaged prints may also be obtained by titration. The weight of

an alkaline printing paste deposited on a fabric is determined by titrating potentiometrically to pH 4.5 both the print and the colour paste. The dye content of the print is calculated from the known dye content of the paste.

W. K. R.

**Accelerated Washfastness Test.** A.A.T.C.C. Tentative Test No. 3A. *Amer. Dyestuff Rep.*, 39, P 657-P 658 (2nd Oct. 1950).

An accelerated laundering test is applicable to cotton or linen fabrics, one 45-min. test being equivalent, in colour loss and abrasive action, to 3 commercial or domestic launderings. Samples are agitated in 50 ml. of 0.2% soap and 0.2% sodium metasilicate solution at 160°F. in metal tubes containing 100 steel balls, up to 20 tubes being mounted at right-angles to a shaft in such a manner that they are given a succession of long throws and strong impacts.

J. W. B.

**Determination of the Fastness to Acid Milling of Dyeings and Prints.** Schweizerischer Verband für die Materialprüfungen der Technik, Kommission 25. Richtlinien-Entwurf A 2572. *Textil-Rund.*, 5, 364 (Sept. 1950).

The test pieces together with white material are treated mechanically in a 40 : 1 liquor containing 2 ml. sulphuric acid 168°Tw. per litre for 1 hr. Two temperatures are employed, viz. 60°C. for light milling and 90°C. for heavy milling. The test pieces are washed off in water, separated, and dried at a max. temp. of 60°C.

B. K.

**Determination of the Fastness to Soda Boiling of Dyeings and Prints.** Schweizerischer Verband für die Materialprüfungen der Technik, Kommission 25. Richtlinien-Entwurf A 2551. *Textil-Rund.*, 5, 365 (Sept. 1950).

The test pieces are bound round a glass rod and treated for 1 hr. at the boil in a 100 : 1 liquor ratio with (a) 5 g. anhydrous sodium carbonate per litre and (b) 5 g. anhydrous sodium carbonate and 4 g. sodium m-nitrobenzenesulphonate or similar product per litre. The liquor ratio must be kept constant by replenishing water lost by evaporation. The test pieces are removed from the rod, washed off, separated, and dried not above 60°C.

B. K.

**Determination of the Fastness to Kier Boiling of Dyeings and Prints.** Schweizerischer Verband für die Materialprüfungen der Technik, Kommission 25. Richtlinien-Entwurf A 2552. *Textil-Rund.*, 5, 366 (Sept. 1950).

The test pieces are covered with grey desized cotton and treated under 0.5 atm. for 4 hr. in a soln. containing 4 ml. caustic soda 35% (sp. gr. 1.38 or 40°Bé.) and 4 g. sodium m-nitrobenzenesulphonate or similar product per litre. The test pieces are washed off, separated, and dried not above 60°C.

B. K.

**Accelerated Aging of [Cotton] Fabric—Effects of Elimination of Sunshine on Weather-exposed Fabric and Subsequent Deterioration in Storage.** J. Maas. *Amer. Dyestuff Rep.*, 39, P 693-P 698, P 714 (16th Oct. 1950).

Cotton sheeting is exposed for different periods to (a) all the weather, with devices to exclude (b) sun and (c) sun and rain. After exposure half the samples are neutralized, and all are stored under conditions of controlled temp. and R.H. Periodic examinations, e.g. of cuprammonium fluidity, breaking strength, and  $H_2SO_4$  equivalent, are made to estimate degradation, and it is found that the non-neutralized samples continue to deteriorate during storage, this being pronounced during the first 4 months and decreasing during a further 4 months. Degradation increases with increasing initial  $H_2SO_4$  equivalent, which drops during storage. Correlation with weather conditions is complex, but factors other than sun have a profound influence, and it is suggested that tests should always be carried out immediately after exposure.

J. W. B.

**New Wool-destroying Fungus—*Ctenomyces* Species.** S. R. S. Gupta, S. S. Nigam, and R. N. Tandan. *Text. Research J.*, 20, 671-675 (Oct. 1950).

A species of *Ctenomyces* having a remarkable capacity for destroying wool has been isolated from damaged wool

material. The morphology and nature of growth of the species have been studied. The first sign of attack of the species on wool is the dissolution of the cementing medium of the scales, leading to their separation from the cortex. The mycelium then penetrates the cortex with separation of the spindle-shaped fibrils. It would appear that only the cementing medium between the cortical cells is attacked and digested, not the cells themselves. It is suggested that the new species could be used as a test organism for the evaluation of rotproofing agents on wool.

P. C.

**Fishy Smell in Goods finished with Urea- or Melamine-Formaldehyde Resins—Detection of Trimethylamine.** W. Kraus. *Textil-Rund.*, 5, 393-399 (Oct. 1950).

Fabrics finished with urea- or melamine-formaldehyde resins may develop a fishy smell, especially under conditions of high humidity and temperature, due to formation of trimethylamine, a method for detecting which has been devised. A 6-g. piece of fabric is boiled, in the presence of melamine and a suspension of mercuric oxide, until a few c.c. of distillate has been formed. The distillate is passed into a solution of iodine in potassium iodide, when trimethylamine, if its concn. exceeds 0.5 mg. per 100 c.c., gives a precipitate of trimethylamine tetraiodide. The amount present may be estimated roughly by collecting the precipitate in a graduated capillary tube. Full experimental details are given for the method, which should be useful for testing the liability of fabrics to develop an odour on storage or during use.

F. A.

**Testing the Resistance of Mothproofing Treatments to Dry Cleaning and Washing.** Schweizerischer Verband für die Materialprüfungen der Technik, Kommission 25. Richtlinien-Entwurf A 3011. *Textil-Rund.*, 5, 413 (Oct. 1950).

Resistance to moths is determined before and after specified dry cleaning and washing treatments.

F. A.

**Colour Reactions for Lignin.** M. S. Bardinskaya. *Doklady Akad. Nauk S.S.R.*, 73, 345-348 (11th July 1950).

The results of two well-known tests for lignin—the reactions with phloroglucinol +  $H_2SO_4$  and with aniline sulphate—and also the colour reactions with sulphuric acid and with 72%  $H_2SO_4$  are tabulated for vanillin, coniferyl alcohol, and other related substances, most of which have been obtained from lignin. The appearance under the fluorescence microscope is also recorded. The results of the same tests, made on the micro-scale on sections of various plants, are tabulated also.

A. E. S.

## PATENTS

**Reflection Photometer.** H. W. Ellis. B.P. 640,691.

In a reflection photometer the material under examination is caused to rotate or otherwise move so that a representative area is continuously scanned by the beam of light during the time of measurement, thus enabling an average value of the reflectivity of the scanned area to be obtained.

C. O. C.

**Measuring Humidity.** Foxboro Co. B.P. 644,675.

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C. O. C.

**Non-destructive Mechanical Test for Animal Fibres [Interaction between Concentrated Neutral Salt Solutions and Wool].** H. M. Burke. (VI, p. 39.)

## XV—MISCELLANEOUS

**Dermatitis and Fabrics.** L. Schwartz. *Penna. Med. J.*, 53, 593-598 (1950); *Chem. Abs.*, 44, 8119 (10th Sept. 1950).

Skin damages caused by synthetic fibres and finishes are reviewed.

C. O. C.

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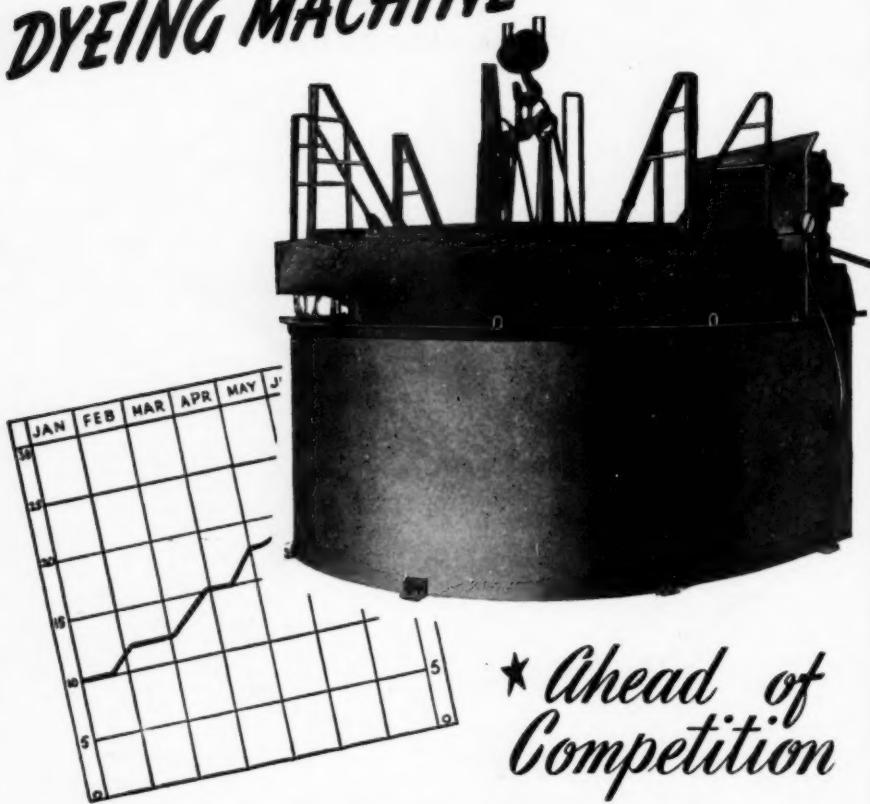
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## BRADFORD JUNIOR BRANCH

*All meetings to be held in the Technical College, Bradford, at 7.15 p.m.*

1951	
Wednesday 24th Jan.	H. R. Hadfield, M.Sc. (I.C.I. Ltd.). <i>The Dyeing of Fast Shades on Nylon</i>
Friday 9th Feb.	A. E. Battye, M.Sc., A.R.I.C., F.T.I. <i>The Location of Polymers in Textile Fibres</i>
Tuesday 27th Feb.	K. Richards, B.Sc., Dip.Ed. Lecture with film strip illustration, <i>The Scientific Story of Wool</i>
Monday 12th March	F. W. Bradley, Esq. (C.A.C. Ltd.). <i>The Application and Properties of Coprantine Dyes</i> . Lecture to be illustrated with many samples
Tuesday 20th March	STUDENTS' NIGHT
Monday 16th April	ANNUAL GENERAL MEETING

## LEEDS JUNIOR BRANCH

*All meetings to be held in the Colour Chemistry Lecture Theatre, Leeds University*

1951	
Tuesday 30th Jan.	Dr. C. H. Giles (Royal Technical College, Glasgow). <i>Colour Photography</i> . 4 p.m.
Tuesday 20th Feb.	Dr. T. Richardson (I.C.I. Ltd.). <i>Dyeing Practice Overseas</i> . 4 p.m.
MANCHESTER JUNIOR BRANCH	
<i>All meetings held in the Reynold's Hall, College of Technology, Manchester, at 6.30 p.m.</i>	

1951	
Tuesday 30th Jan.	F. W. Bradley, Esq., of Clayton Aniline Co. Ltd. <i>The Application of Metallizable Dyes</i>
Tuesday 3rd April	S. R. Cockett, M.Sc.(Tech.), F.R.I.C., of the Bradford Dyers' Association. <i>Detection and Correction of Faults in Dyed Materials</i>

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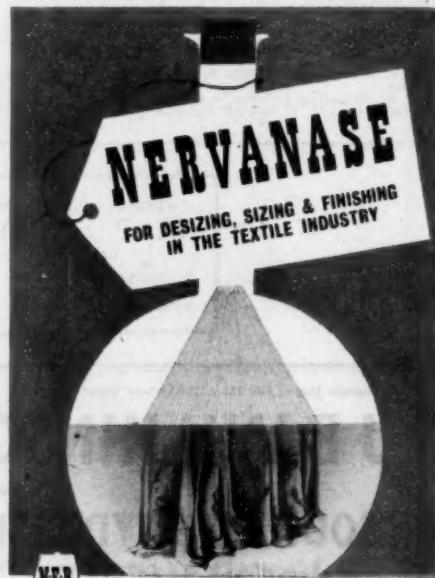
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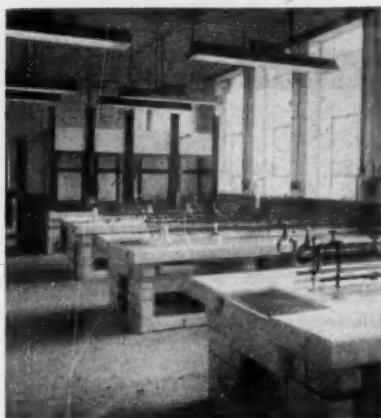


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